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INDUSTRIAL WASTE DIVERSION PROGRAM

ASSESSMENT OF ALTERNATIVE PHENOL RECOVERY PROCESSES

PHASE II

PILOT SCALE TESTING

AUGUST 1991

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PIBS 1590

ASSESSMENT OF ALTERNATIVE PHENOL RECOVERY PROCESSES PHASE II PILOT SCALE TESTING

Report prepared for:

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AND

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1.0 SUMMARY AND CONCLUSIONS

CanadianOxy produces phenol-formaldehyde resins at its Fort Erie plant. The manufacturing process involves a polymerization reaction in which water is generated as a by-product. Excess water is distilled from each of two batch reactors, condensed and collected in separate distillate receivers. About 1,000 gals of distillate are collected each day generally containing 5 to 8% phenol. The distillate is presently being hauled off-site for disposal. In 1987, haulage and disposal costs were in the order of $280,000 and the value of lost phenol was estimated at $120,000. CanadianOxy is now considering the feasibility of recovering phenol from the distillate and treating the effluent on-site to meet sewer discharge limits.

ZENON was contracted by CanadianOxy in July, 1988 to conduct a two-phase study aimed at evaluating existing phenol recovery technologies, selecting the most feasible process for the application and confirming the viability of the selected process with bench and pilot scale testing. In Phase I of the study, resin adsorption with formaldehyde regeneration was selected as the most suitable technology after a review of seven possible processes. The technical and economic feasibility of the process was demonstrated first by bench-scale tests and a preliminary economic assessment. A pilot-scale evaluation of the process was carried out in Phase II of the study, which started in June, 1989. This report presents the results of the Phase II work and a revised economic assessment of the process.

The following conclusions and recommendations are made based on the Phase II study:

- Considering the differences in equipment and test conditions, bench-scale operating data were reproduced reasonably well at the pilot scale.
- Although phenol adsorption rates were observed to increase with increasing operating temperatures between 30 and 50°C, the effect on the overall adsorption capacity of the resin

bed was found to be insignificant for bed depths greater than 1 m. Since the optimal bed depth would be greater than 1 m for a full-scale system, an operating temperature between 25 and 30°C is recommended for the full-scale process.

- The elution process was observed to be more sensitive to changes in operating temperature than the loading process. An operating temperature between 50 and 60°C was recommended for the elution step due to the improved elution efficiency at elevated temperatures and the fact that formaldehyde is currently being stored at 60°C.
- Tests over a range of bed depth and hydraulic loading combinations indicated that a hydraulic loading less than 4 m/h and bed depth of at least 0.6 m should be employed to maximize resin bed utilization.
- Mass transfer rates appeared to be more pore-diffusion-controlled in the loading step and more film-diffusion-controlled in the elution step for hydraulic loadings ranging from 2 to 5 m/h. As a result, adsorption capacity was fairly independent of hydraulic loading, while elution efficiency improved significantly with increases in hydraulic loading between 2 and 4 m/h. Although a hydraulic loading of 4 m/h was selected for the elution step in long-term tests, higher hydraulic loadings may have led to some further improvement in elution efficiency.
- In long-term tests, phenol recovery levels ranged from 75% to 89%. Higher phenol recovery levels were achieved with distillate feeds containing higher phenol concentrations.
- After elution with formaldehyde, about 3 bed volumes of fresh water were generally required at a hydraulic loading of 10 m/h to reduce effluent formaldehyde concentrations below 1%. To eliminate disposal problems, this effluent can be used to dilute 50% formaldehyde feed down to 40% for use in the recovery process.
- The wide fluctuations observed in distillate concentration (3.5 - 8.3%) and pH (2.5 - 5.8) had a significant effect on phenol concentrations in both the treated distillate stream and eluate stream for recycle. As a result, measures will have to be taken in a full-scale recovery system

to control the effluent phenol concentrations. Possible strategies are either the installation of a distillate equalization tank or a feedback control system involving on-line monitoring of distillate and effluent phenol concentrations.

- The capital investment and annual operating cost for the phenol recovery process were estimated at $449,000 and $110,000 respectively if distillate is biologically treated after phenol recovery. Taking credit for recovered phenol and savings in off-site disposal costs, a payback period of 4.5 years was calculated.
- This payback period is longer than the 1.8 year period that was calculated from the results of the bench scale test work and preliminary economic assessment completed in Phase I. The primary reasons for this are as follows:
  - No storage tank requirements were included in the Phase I capital cost estimate.
  - The estimated annual operating cost increased by 47% in this analysis compared to that estimated in the Phase I study, mainly due to the inclusion of sludge disposal costs and significantly higher operating and maintenance cost estimates for the biological treatment unit.
  - Credits taken for recovered phenol and savings in off-site distillate disposal costs were about 23% lower in this analysis than in the Phase I study due to slumping phenol prices and the lower disposal costs currently paid by CanadianOxy.
- For an increase in distillate production from 1,600 tonnes/year to 2,100 tonnes/year, the payback period on investment would be lowered to 1.7 years.
- A biological treatment plant to treat distillate after phenol recovery should accompany the installation of a recovery system in order to realize attractive returns on capital investment. The payback period would be increased from 1.7 to 4.7 years if distillate continues to be disposed of off-site after installation of a recovery system.

2.0 INTRODUCTION

2.1 Background

CanadianOxy Chemicals Ltd. owns and operates a phenol-formaldehyde plant in the City of Fort Erie, Ontario. The plant produces about 12 million pounds per year of moulding compounds, industrial resins and foundry resins.

The manufacturing process involves reacting phenol and formaldehyde in two batch processing kettles. The condensation polymerization reaction produces a liquid resin which is cooled and flaked on a flaker belt. The flaked product is either sold directly, pulverized and sold, or used in-house in pulverized form to produce moulding compounds.

Water is produced as a by-product of the polymerization reaction. The water vapour leaving each kettle is condensed and collected in a distillate receiver. Throughout a batch operation, the distillate receivers are emptied three times producing three distinct distillate "cuts". The first cut contains approximately 0.5-1% phenol and the second cut between 2 and 4% phenol. The third cut separates into two distinct phases, a saturated water phase containing approximately 8% phenol and a heavier organic phase containing about 72% phenol. In all cases, the phenol present in the distillates is primarily the same simple phenol used as raw material for the resin production process. The phenol levels in the distillates are however too low for it to be reused directly in the reaction without first being concentrated.

At the present time, distillate from both kettles is shipped off-site for treatment at Nova in Sarnia. In 1987, CanadianOxy hauled approximately 350,000 gallons of distillate off-site (approximately 1,000 gallons per day) along with an additional 44,000 gallons (1,000 gallons per week) of phenol contaminated dyke water containing up to 500 mg/L of phenol. The total cost for disposing of this wastewater was in the order of $250,000 to $300,000 per year. In addition, the value of the phenol content in the disposed distillate was about $120,000. Therefore, the potential net saving was about $400,000 per year if all of this phenol could have been recovered for reuse and the effluent discharged to the municipal sewer after biological treatment.

CanadianOxy is converting some of their existing process to a "low free phenol process" similar to that which has been used in Europe for a number of years. This change will have potentially far ranging and positive environmental benefits to the end users of phenol-formaldehyde resins by significantly reducing the tendency for the resins to release phenol to the environment. However, a disadvantage of the low free phenol process is that it will significantly increase the phenol concentration in each of the three distillate cuts. Therefore, in order for CanadianOxy to take advantage of low free phenol technology and maintain the plant's operating efficiency, measures will have to be taken to recover and reuse phenol within the plant to the maximum extent possible.

ZENON was contracted by CanadianOxy in the summer of 1988 to carry out a two phase study aimed at evaluating existing phenol recovery technologies, selecting the most feasible process for the application and confirming the viability of the process with bench and pilot scale testing. Phase I of the study involving a technology review and bench-scale testing of a selected recovery process was completed in January, 1989. After reviewing seven potential recovery processes, it was concluded that resin adsorption with formaldehyde regeneration was the only process capable of recovering phenol economically at the Fort Erie plant. A bench-scale evaluation of the resin adsorption process confirmed its technical feasibility, and a preliminary economic analysis suggested that a payback period of as little as 1.8 years was possible.

In light of the potentially favourable economics for the recovery process, ZENON was allowed to proceed with the second phase of the project involving pilot-scale testing. A resin adsorption pilot plant was designed and constructed by ZENON and sent to the Fort Erie plant in August, 1989. Pilot scale testing got underway in January, 1990 and was completed in July, 1990. This report presents the results of the pilot study, ZENON's interpretation of the results and possible design specifications for a full scale recovery system. A more detailed cost analysis of the full-scale resin adsorption process is also provided.

2.2 The Resin Adsorption Process

Resin adsorption works on the same principles as carbon adsorption, i.e. molecules are selectively attracted from a fluid phase and bonded by a combination of physical and chemical forces to the surface of a solid phase, commonly termed the adsorbent. Generally, non-polar or hydrophobic solutes are adsorbed onto non-polar adsorbents while polar or hydrophilic solutes are adsorbed onto polar adsorbents. The phenol molecule consists of a hydrocarbon ring with a polar hydroxyl group attached. The molecule therefore has both hydrophilic and hydrophobic "ends" and can be adsorbed onto either non-polar or slightly polar adsorbents.

For phenol to be effectively adsorbed in this application, the affinity of the adsorbent surface for phenol must be greater than the solvating forces exerted by the aqueous phase. Likewise, for the elution of adsorbed phenol, the solvating forces exerted by the selected eluant must overcome the attractive forces of the adsorbent.

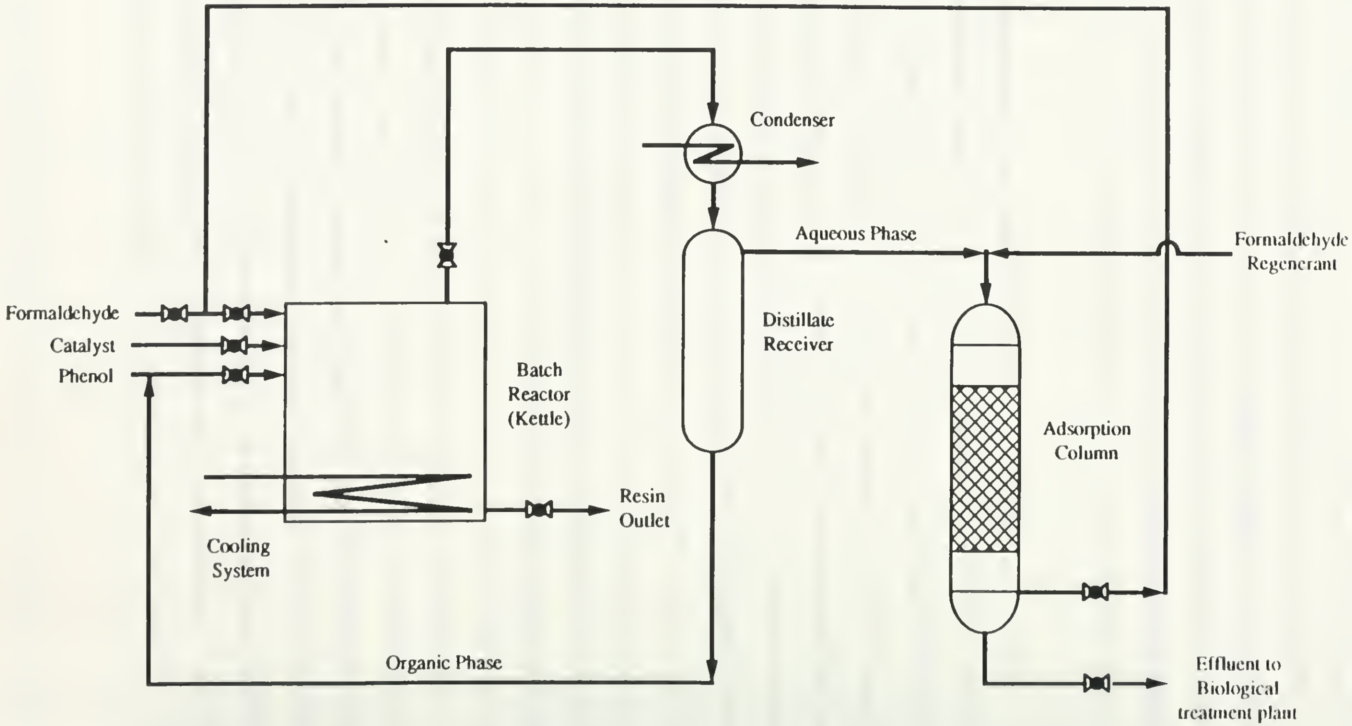
Besides polarity, other factors have to be considered in selecting the most suitable adsorbent for an application. These factors include surface area, pore size, chemical and structural stability, and hydraulic characteristics for column operation. The importance of these factors is discussed extensively in the literature (Weber, 1972).

Macroreticular resin adsorbents have an advantage over activated carbon in that their adsorption forces are much weaker, allowing them to be effectively regenerated with suitable solvents. This provides an opportunity for recovering sorbed materials for reuse. Another advantage of resin adsorbents is their structural integrity which permits them to be reused for hundreds of operating cycles with very little loss in bed capacity.

Based on bench scale tests, the Amberlite XAD7 adsorption resin manufactured by Rohm & Haas was selected for pilot scale tests. The XAD7 resin is a non-ionic adsorbent with intermediate polarity. More information on the resin can be found in the manufacturer's literature provided in Appendix D. Since it contains both hydrophobic and hydrophilic surfaces, the resin will adsorb either the hydrophobic or hydrophilic end of the phenol molecule. Because of this feature, the adsorption capacity of XAD7 is not significantly affected over the pH range of 2 to 8 typically observed in CanadianOxy's distillates. Phenol is a weak acid and dissociates in water to an extent dependent on pH. When dissociated, the polarity of the phenol molecule is increased over its non-dissociated state resulting in little or no adsorption onto hydrophobic or non-polar surfaces. The dissociated molecule can however still be adsorbed on the polar surface of the XAD7 resin, thereby reducing the effect of pH.

The simplified flow diagram presented in Figure 2.1 illustrates how resin adsorption can be employed to recover phenol at the CanadianOxy plant. Water vapour drawn off the batch reactor during the phenol formaldehyde polymerization reaction is condensed and collected in a distillate receiver. The distillate also contains some phenol stripped from the liquid mass in the reactor, but virtually no formaldehyde since it is the limiting reactant. After the third distillate cut is received, the phenol concentration in the disillate exceeds its solubility (approximately 8%) and an organic phase containing about 72% phenol settles to the bottom of the receivers. This phenol rich phase

Figure 2.1 - Process Flow Diagram of Phenol Recovery System



will be taken off the bottom of the receiver and recycled back to the reactor after some intermediate storage.

The aqueous phase of the distillate containing 4 to 8% phenol will be pumped from the receiver to the resin adsorption system. The treated effluent containing less than 1% phenol will be directed to a bio-oxidation process for further treatment prior to discharge to a municipal sewer. Once the adsorption capacity of the resin bed is exhausted, the column will be regenerated with a 40% formaldehyde solution (also used as a feedstock to the batch reactor). The eluant containing an average of 4 to 12% phenol is recycled as a feed to the reactor.

2.3 Objectives of Pilot Study

The overall objective of the Phase II work was to operate a pilot-scale test unit on-site for a minimum of three months to confirm the technical and economic suitability of the resin adsorption process for recovering phenol from distillate feeds. More specific objectives were:

- To design, construct and install a pilot-scale resin adsorption process for recovering phenol at the CanadianOxy plant.
- To reproduce XAD7 resin breakthrough and regeneration profiles on a pilot-scale for comparison with results from bench-scale column tests.
- To optimize process performance by operating the plant under varying operating conditions.
- To conduct longer-term testing to establish the reliability and flexibility of the recovery process.
- To perform a more detailed economic assessment of the phenol recovery process from the pilot-scale test data.
- To provide possible design specifications for a full-scale phenol recovery process.

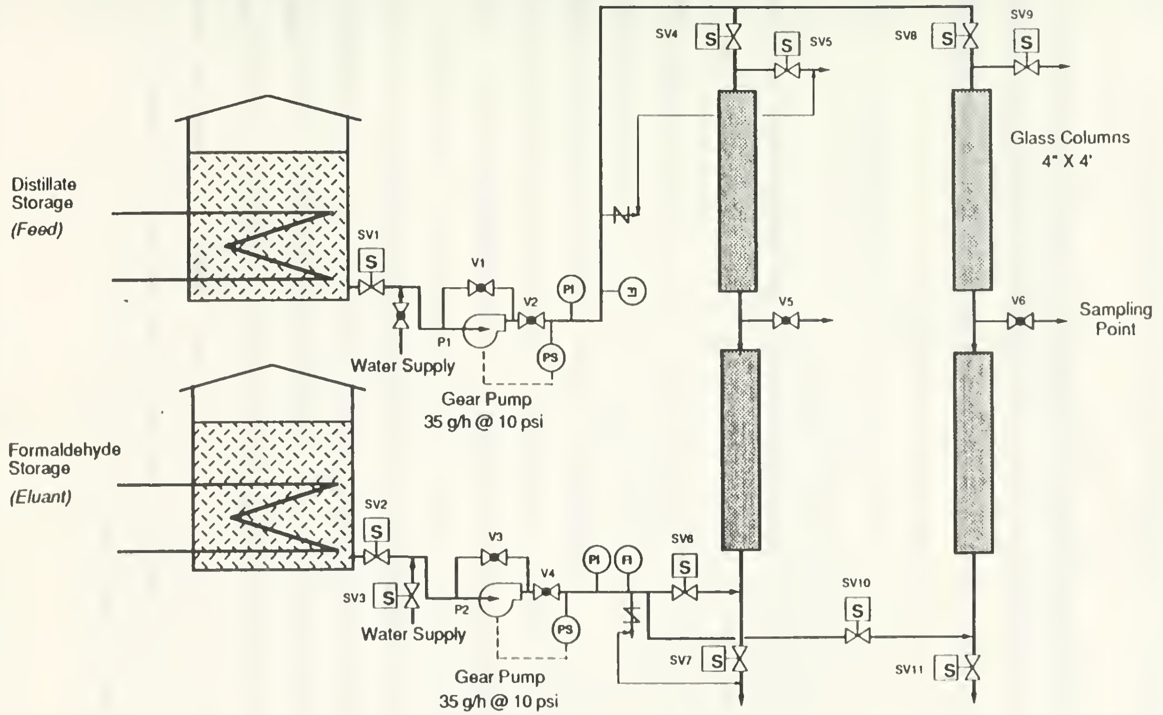
3.0 TEST EQUIPMENT AND PROCEDURES

3.1 Description of Pilot Plant

The resin adsorption pilot unit was designed and constructed by ZENON Environmental Inc. to carry out a pilot-scale evaluation of the process for recovering phenol at the CanadianOxy plant. The system consists primarily of four glass columns 120 cm high and 10 cm in diameter, two process pumps, instrumentation, an automatic control system, tubing, valves and other fittings. The entire system is skid-mounted and measures 150 cm in length, 82.5 cm in width and 155 cm in height. A process and instrumentation diagram for the pilot unit is presented in Figure 3.1. Wiring diagrams and the control panel layout for the unit can be found in Appendix C.

At the heart of the automatic control system is a programmable logic controller (PLC). The PLC allows the system to automatically run through a six-step operating sequence that simultaneously loads and regenerates alternating column sets. Alarm conditions triggered by pressure and temperature switches on the pump discharge lines are also processed by the PLC before tripping an audible alarm and panel lights and, in some cases, shutting off the pumps. The control narrative for the pilot unit is described in a separate document entitled "Pilot Plant Operating Procedure and Detailed Workplan" supplied to CanadianOxy in September, 1989.

Materials of construction were carefully selected to withstand long-term exposure to saturated phenol and formaldehyde solutions. All solenoid and manual valves are constructed of 316 stainless with viton seats. Other fittings are made of either 316 stainless or polypropylene. The feed lines to both process pumps consist of 316 stainless tubing while high density polyethylene tubing was used for connecting the four columns.



LEGEND

PI	Pressure Indicator
FI	Flow Indicator
PS	Dual Pressure Switch (high/low)
S	Solenoid

1	Jun 15			JMB
REVISION#	DATE	DESCRIPTION	BY	APPROVED

SCALE none	DRAWN J M Benko DATE April 10 1989	CHK'D J M Benko DATE April 10 1989	APP'D J M Benko DATE April 10 1989
CUSTOMER			CONTRACT# C08 N44
TITLE	Process & Instrumentation Diagram for Phenol Recovery Pilot Plant		DRAWING# Figure 11

The use of flexible tubing between columns allowed the system to be easily replumbed to provide different flow configurations. By activating the PLC operating sequence for only one set of columns, the system can be operated with up to four columns in series.

Auxiliary equipment provided by CanadianOxy included a 1,000 L plastic tank for storing distillate, 200 L plastic drums for storing formaldehyde and collecting effluents, and electrical drum heaters for preheating distillate and formaldehyde feeds. The storage tanks were connected to the pilot unit with polyethylene tubing and rubber hose.

3.2 General Operating Procedures

A description of the six-step operating sequence executed by the PLC is presented below. Details on system start-up, shut down and other operating procedures were provided in the "Pilot Plant Operating Procedure and Detail Workplan" document previously supplied. Note that the system was only operated in an automatic mode for multi-cycle runs done towards the end of the pilot study. Only one set of columns was activated during these tests (rather than both sets for continuous distillate treatment) to minimize labour and analytical requirements. Figures 3.2 to 3.7 illustrate the flow paths associated with each of the six operating steps.

Step 0 - Loading of Column Set #1 and Elution of Column Set #2

Distillate feed is pumped through columns 1a and 1b. Phenol is adsorbed from the aqueous medium onto the resin bed thereby producing a column effluent with a much reduced phenol concentration. Meanwhile, formaldehyde eluant is simultaneously pumped counter-currently through columns 2b and 2a. The effluent or eluate stream from column set #2 contains high concentrations of phenol dissolved in the formaldehyde solution and will eventually be recycled as feed to the polymerization reactors.

Active: Solenoid valves SV1, SV2, SV4, SV7, SV9 and SV10, and pumps P1 and P2.

Step 1 - Loading of Column Set #1 and Displacement of Eluant Void in Column Set #2

Distillate feed continues to be pumped through columns 1a and 1b. Meanwhile, the elution step for column set #2 is completed, and tap water is fed through these columns to displace the eluant void before making the column available for a next loading step.

Active: Solenoid valves SV1, SV3, SV4, SV7, SV9 and SV10, and pumps P1 and P2.

Step 2 - Loading of Column Set #1 and Column Set #2 in Standby

Distillate feed continues to be pumped through columns 1a and 1b. Meanwhile, column set #2 is prepared for a next loading step and is put on standby until the beginning of step 3.

Active: Solenoid valves SV1, SV4 and SV7, and pump P1.

Step 3 - Loading of Column Set #2 and Elution of Column Set #1

Distillate feed is pumped through columns 2a and 2b. Phenol is adsorbed from the aqueous medium onto the resin bed thereby producing a column effluent with a much reduced phenol concentration. Meanwhile, formaldehyde eluant is simultaneously pumped counter-currently through columns 1b and 1a. The effluent or eluate stream from column set #1 contains high concentrations of phenol dissolved in the formaldehyde solution and will eventually be recycled as feed to the polymerization reactors.

Active: Solenoid valves SV1, SV2, SV5, SV6, SV8 and SV11, and pumps P1 and P2.

Step 4 - Loading of Column Set #2 and Displacement of Eluant Void in Column Set #1

Distillate feed continues to be pumped through columns 2a and 2b. Meanwhile, the elution step for column set #1 is completed, and tap water is fed through these columns to displace the eluant void before making the column available for a next loading step.

Active: Solenoid valves SV1, SV3, SV5, SV6, SV8 and SV11, and pumps P1 and P2.

Step 5 - Loading of Column Set #2 and Column Set #1 in Standby

Distillate feed continues to be pumped through columns 2a and 2b. Meanwhile, column set #1 is prepared for a next loading step and is put on standby until the sequence starts again from step 0.

Active: Solenoid valves SV1, SV8 and SV11, and pump P1.

Figure 3.2 - Loading of Column Set #1 and Regeneration of Column Set #2 (Step 0)

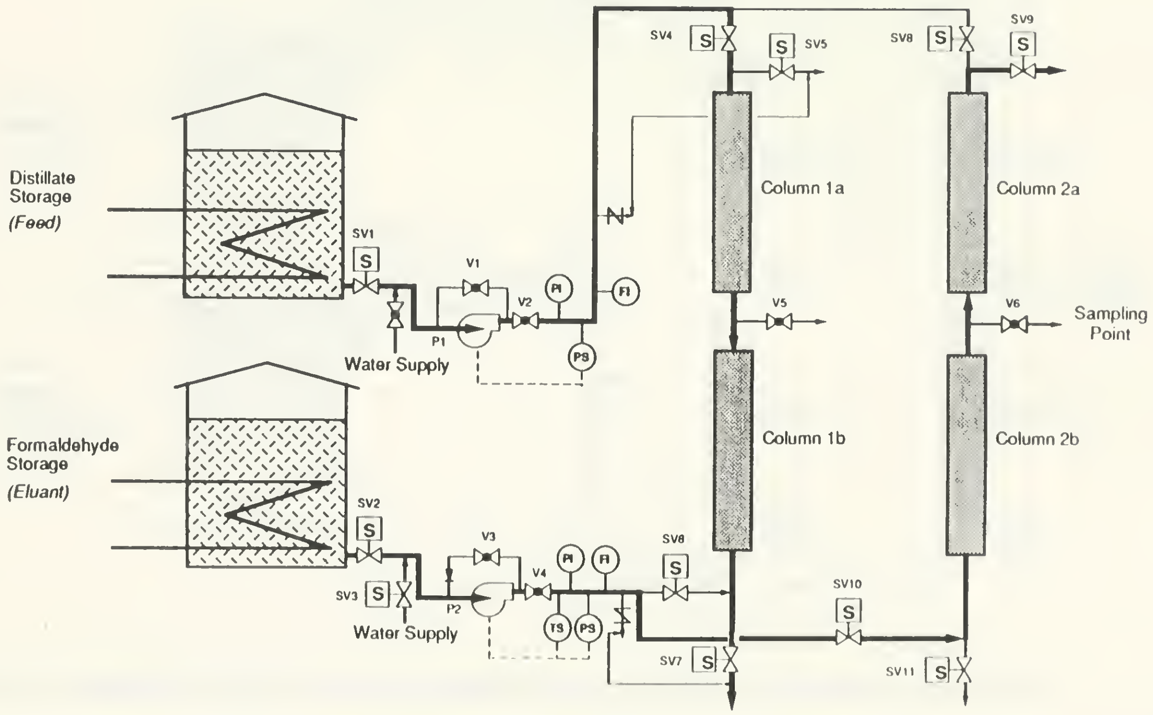


Figure 3.3 - Loading of Column Set #1 and Displacement of Eluant Void in Column Set #2 (Step 1)

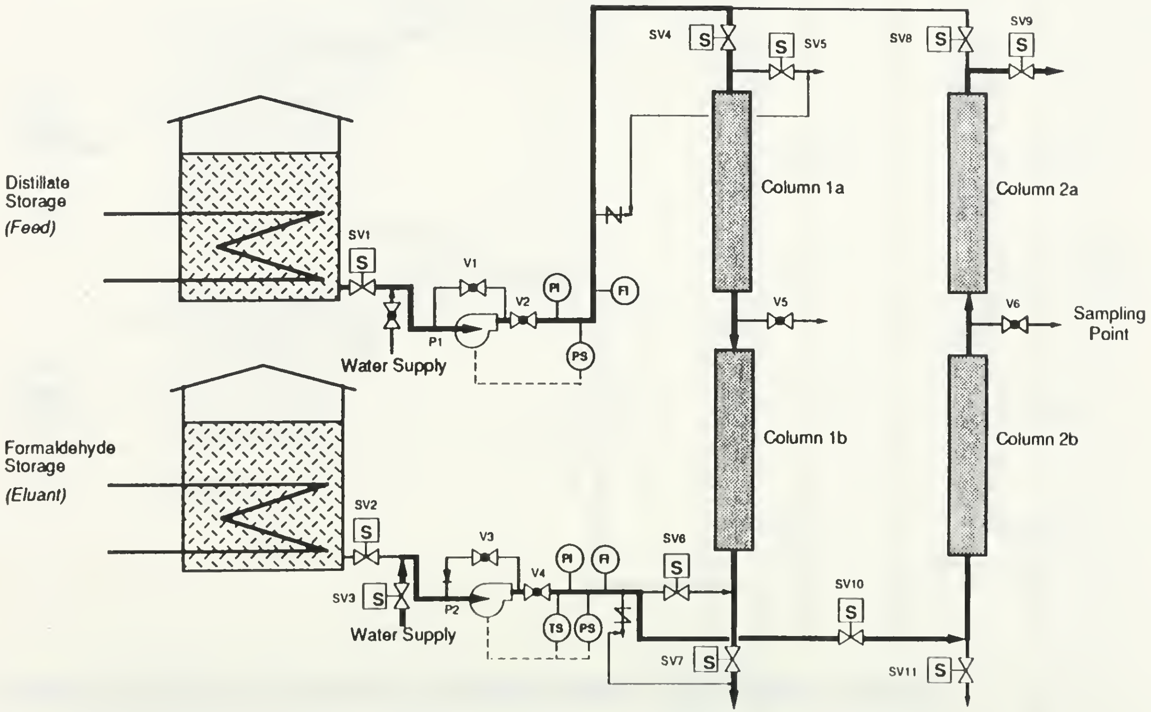


Figure 3.4 - Loading of Column Set #1 and Column Set # 2 in Standby (Step 2)

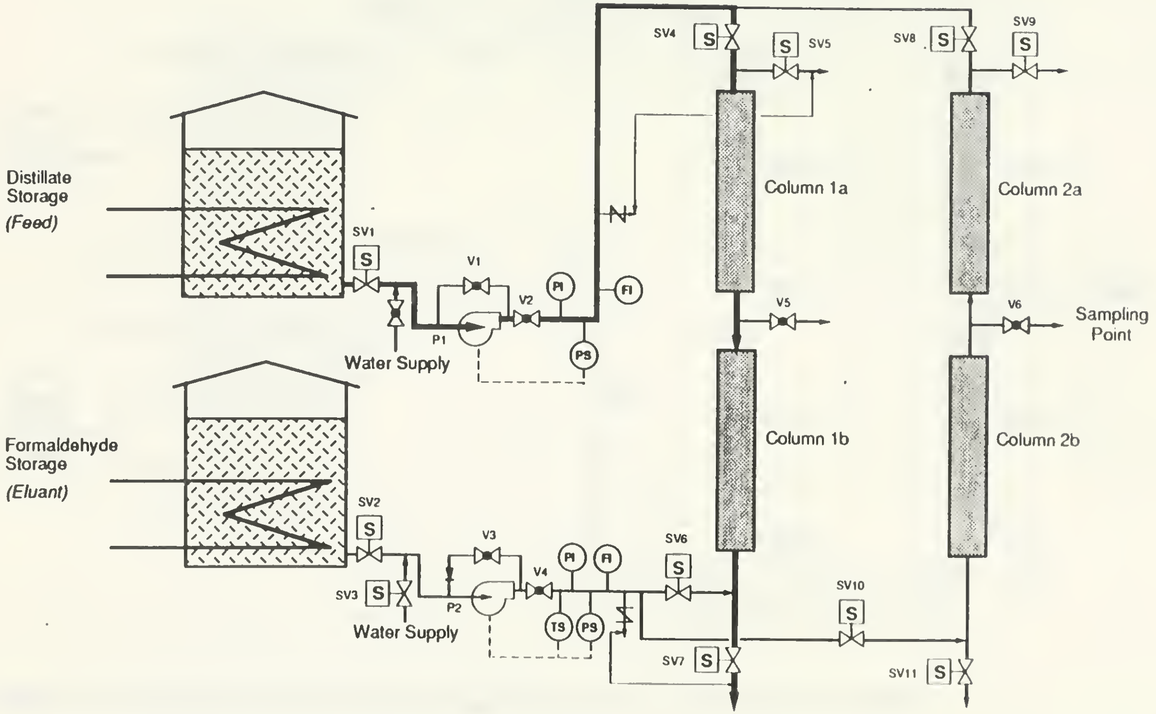


Figure 3.5 - Loading of Column Set #2 and Regeneration of Column Set #1 (Step 3)

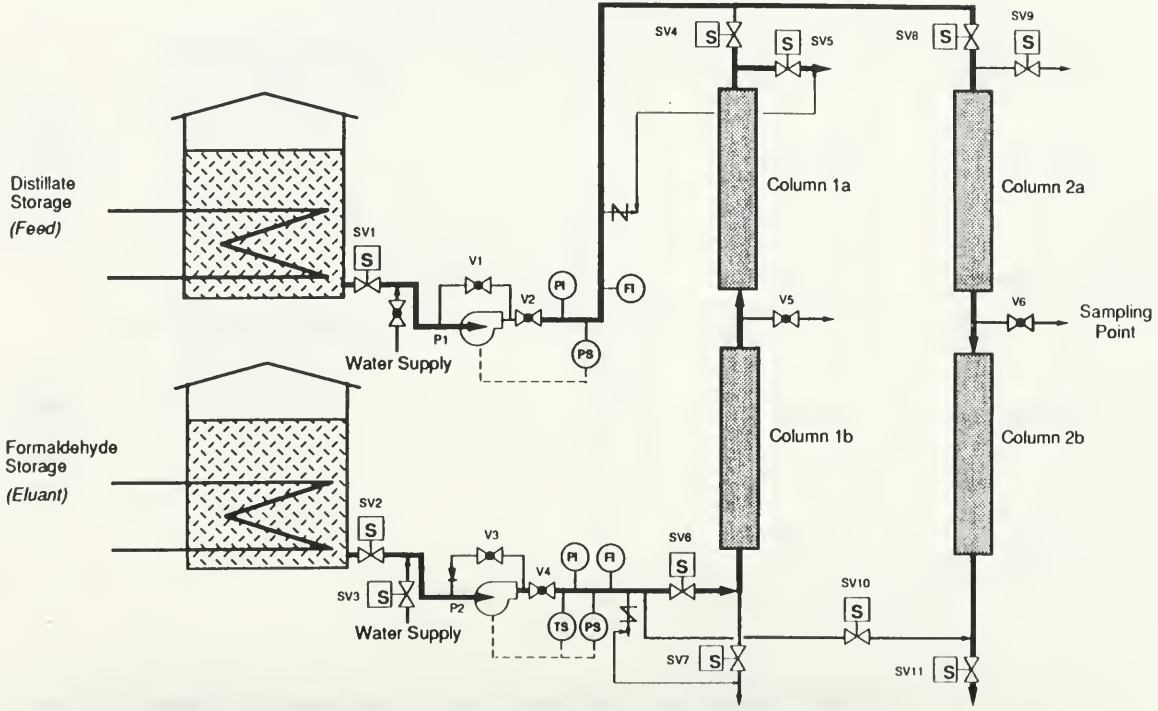


Figure 3.6 - Loading of Column Set #2 and Displacement of Eluant Void in Column Set #1 (Step 4)

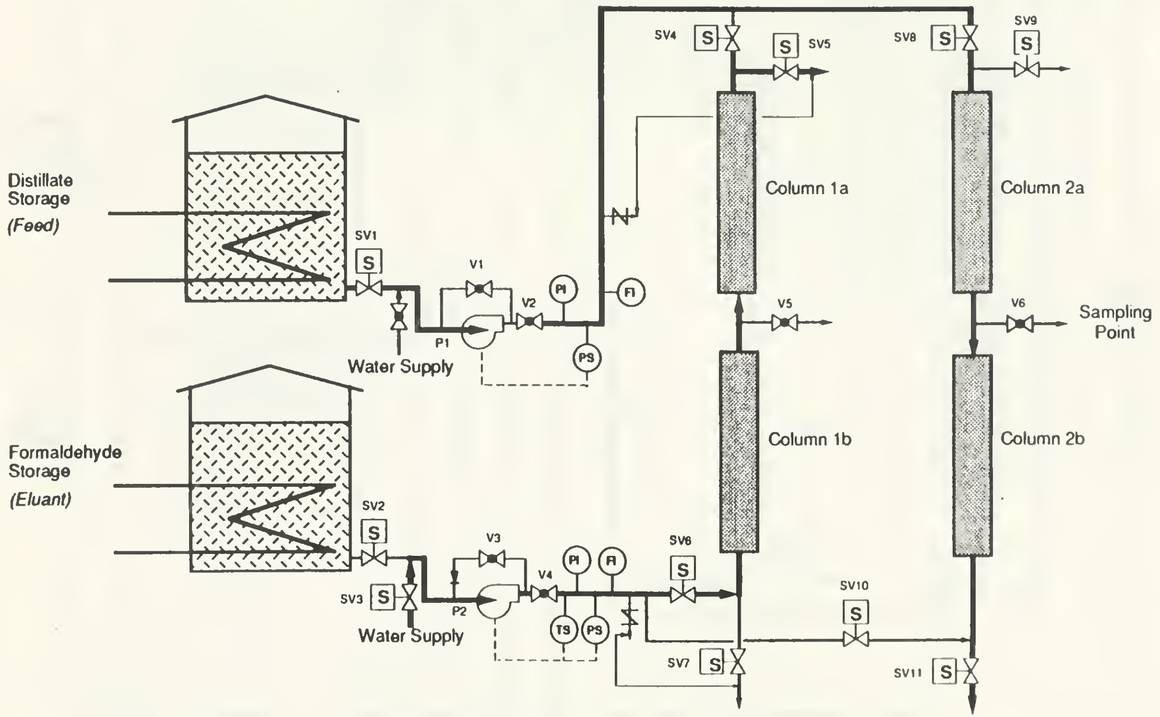
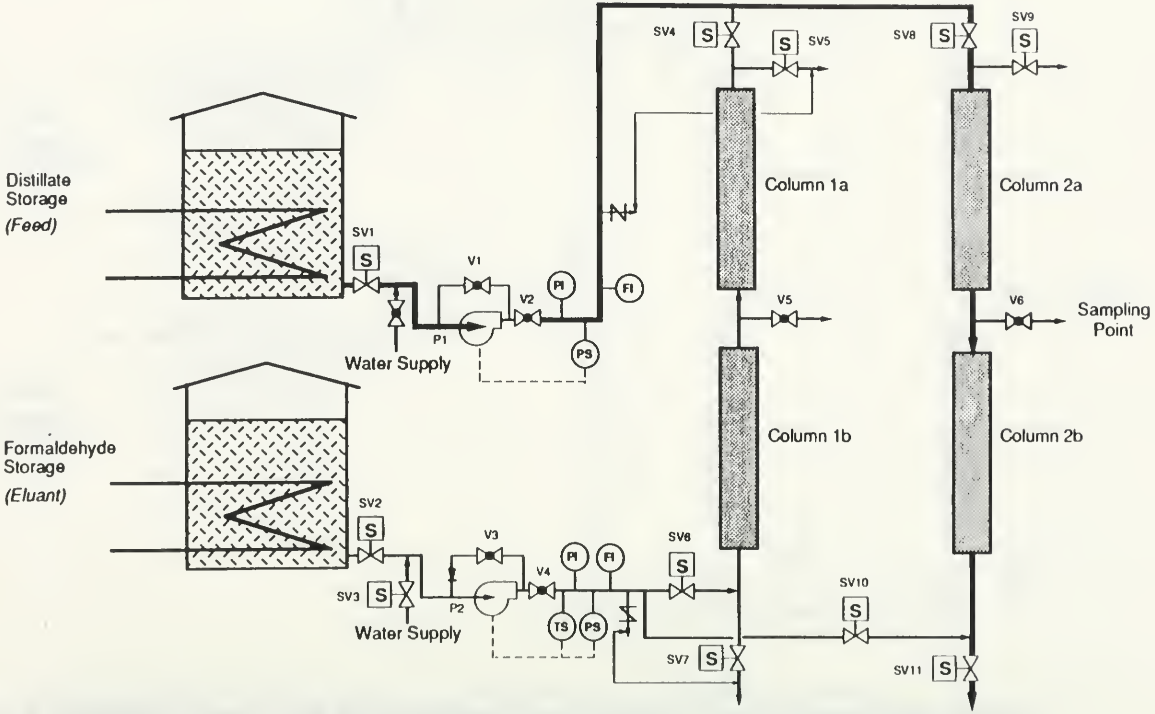


Figure 3.7 - Loading of Column Set #2 and Column Set #1 in Standby (Step 5)



3.3 Pilot Scale Tests

The experimental plan for the pilot study consisted of the following six tasks:

1. Verification of bench-scale results
2. Investigating the effect of distillate temperature on bed capacity
3. Investigating the effect of eluant temperature on elution efficiency
4. Investigating the effect of hydraulic loading and bed depth on bed capacity
5. Investigating the effect of hydraulic loading on elution efficiency
6. Long-term evaluation of the phenol recovery process

A copy of the experimental plan detailing test objectives and procedures can be found in Appendix A.

3.4 Analytical Methods

Phenol analyses were conducted by three different methods. All distillate feed and effluent samples were analyzed by refractive index. In this procedure, phenol concentrations were interpolated from a calibration curve constructed with pure component phenol solutions. The method is routinely used by CanadianOxy to give quick determinations of phenol concentrations in the 0.1% - 8% (w/w) range and has a minimum detection limit of 0.05% or 500 mg/L.

Due to formaldehyde interference, the refractive index method could not be used for determining phenol concentrations in the eluate stream. These determinations were made using a Chemets kit. The Chemets analysis is based on the 4-aminoantipyrine method for analyzing phenol in water and wastewaters. Concentrations ranging from 1 to 10 mg/L can be determined with the Chemets kit with an accuracy of ± 5% and minimum detection level of 0.1 mg/L.

To verify the analytical results reported by CanadianOxy using the above analytical methods, representative distillate feed and effluent samples and an eluate sample were analyzed for phenol at ZENON's laboratories using the standard 4-aminoantipyrine colorimetric method (APHA, 1989). The results of these analyses are compared with values reported by CanadianOxy in Table 3.1. Conductivity values for each sample are also presented in this table. ZENON's analytical technique covers phenol concentrations ranging from 0.001 mg/L to 0.250 mg/L with a sensitivity of 0.001 mg/L.

Some formaldehyde analyses were done on the rinse effluents to determine the extent of formaldehyde removal from the resin bed. A titration method was employed to determine the formaldehyde concentrations. A sodium sulphite solution is added to a fixed sample volume and titrated with hydrochloric acid to a pH end point. Sodium sulphite reacts with the formaldehyde to produce sodium hydroxide. The accuracy of this method is generally within ± 0.5%.

Table 3.1 - Comparison of Analytical Results by Different Methods

Sample Identification	Conductivity * (µS/cm)	Phenol Concentrations (g/L)			Analytical Deviation (%)
		Refractive Index	Chemets Kit	4-aminoantipyrine *	
L3 - Distillate	890	7.7	-	7.8	1%
L3 - 10:00	1230	0.8	-	0.67	-19%
R3 - 5:30	170	-	0.4	0.40	0%

* *Values reported by Zenon Environmental Laboratories*

4.0 RESULTS AND DISCUSSION

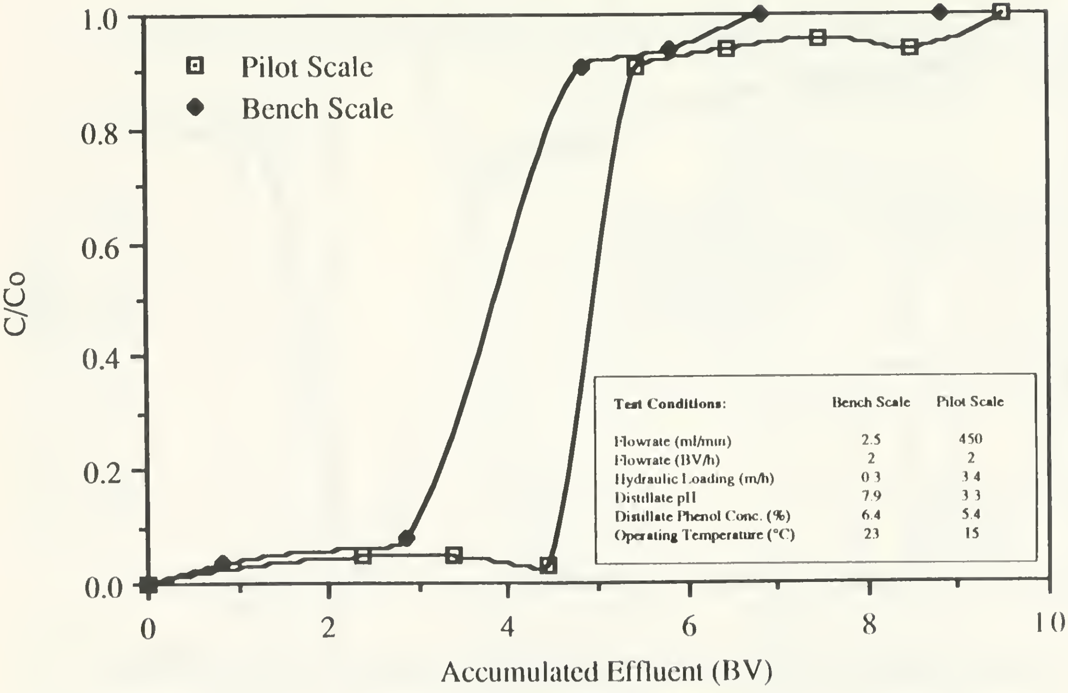
4.1 Verification of Bench Scale Results

The purpose of the initial pilot plant run was to reproduce on a pilot-scale the phenol adsorption capacity and elution efficiency observed with the Amberlite XAD7 resin in bench-scale tests. The pilot-scale test was conducted with the same empty bed contact time (EBCT) as in the bench-scale test, but with significantly different distillate phenol concentrations, distillate pH, operating temperatures and hydraulic loadings. In addition the pilot system was regenerated in a countercurrent mode as opposed to the cocurrent mode employed in the corresponding bench-scale test. Breakthrough and elution profiles for the bench and pilot scale runs are presented in Figures 4.1 and 4.2 along with a tabulation of the relevant test conditions for each case.

Differences in operating temperature and hydraulic loading were expected to have negligible effect on process performance compared to the other process variables. Mass transfer is only marginally improved with a rise in operating temperature from 15 to 23°C. This fact was demonstrated in temperature effect studies (see Section 4.2). Generally, mass transfer in column operations involving packed beds of microporous media (such as carbon adsorption, ion exchange and resin adsorption) is pore-diffusion-controlled and independent of normal hydraulic loadings (Weber, 1972).

Being pore-diffusion-controlled, the resin adsorption process is expected to be most sensitive to the EBCT; this was later demonstrated in other pilot runs (see Sections 4.3 and 4.4). Therefore, by holding this variable constant in the bench and pilot tests, a good indication of the scale-up accuracy could be obtained for the resin adsorption process. In the loading step, breakthrough profiles were observed to be relatively comparable, with 50% breakthrough occurring after 3.9 and 4.9 bed volumes for the bench and pilot scale runs respectively. The earlier breakthrough observed in the bench-scale run is likely the result of the higher phenol loading to the columns, the

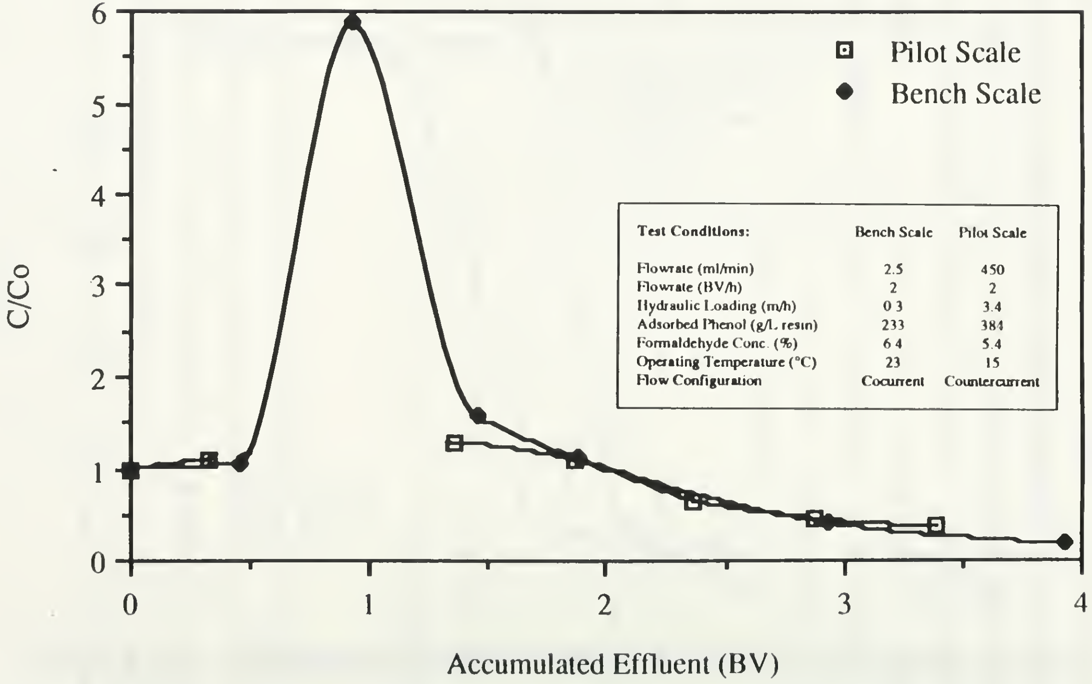
Figure 4.1 - Comparison of Bench and Pilot Scale Breakthrough Profiles



Test Conditions:	Bench Scale	Pilot Scale
Flowrate (ml/min)	2.5	450
Flowrate (BV/h)	2	2
Hydraulic Loading (m/h)	0.3	3.4
Distillate pH	7.9	3.3
Distillate Phenol Conc. (%)	6.4	5.4
Operating Temperature (°C)	23	15

C: Phenol concentration in eluate
Co: Distillate phenol concentration
BV. Bed volume

Figure 4.2 - Comparison of Bench and Pilot Scale Elution Profiles



Test Conditions:	Bench Scale	Pilot Scale
Flowrate (ml/min)	2.5	450
Flowrate (BV/h)	2	2
Hydraulic Loading (m/h)	0 3	3.4
Adsorbed Phenol (g/L resin)	233	384
Formaldehyde Conc. (%)	6 4	5.4
Operating Temperature (°C)	23	15
Flow Configuration	Cocurrent	Countercurrent

C: Effluent phenol concentration
Co: Distillate phenol concentration
BV Bed volume

lower overall adsorption capacity of the resin bed due to the lower percent utilization (compared to equilibrium capacity, a factor commonly termed the "bed depth effect") and the higher distillate pH. Although not as significantly as with less polar adsorbents, the adsorption capacity of the XAD7 resin is expected to decrease somewhat with increasing distillate pH.

The elution profiles shown in Figure 4.2 compare fairly well, indicating a 50% reduction of initial phenol concentration after 2.7 bed volumes of formaldehyde solution for both bench and pilot scale runs. Note that the first half of the elution profiles do not accurately represent changes in eluate phenol concentration with time because of the limited number of data points available. No concentration peak was observed in the pilot scale profile although one likely occurred between 0.5 and 1.5 bed volumes of accumulated eluate.

Elution in a countercurrent configuration generally produces concentration peaks that are sharper and occur earlier than those obtained in a cocurrent configuration. This occurs since the eluate exits the end of the column that is loaded most heavily with phenol. The profiles in Figure 4.2 do not give a good indication of the merits of countercurrent regeneration because of the differences in test conditions.

4.2 Effect of Operating Temperature on Process Performance

The effect of operating temperature on phenol removal in the loading step and elution efficiency in the regeneration step were investigated in a series of four runs (Task 2.3, Runs #1-4 and Task 2.4, Run #1).

Breakthrough and elution profiles at different operating temperatures are presented in Figures 4.3 and 4.4. Empty bed contact times, hydraulic loadings and bed depths were kept constant in all of these runs.

Figure 4.3 - Phenol Breakthrough Profiles at Different Operating Temperatures

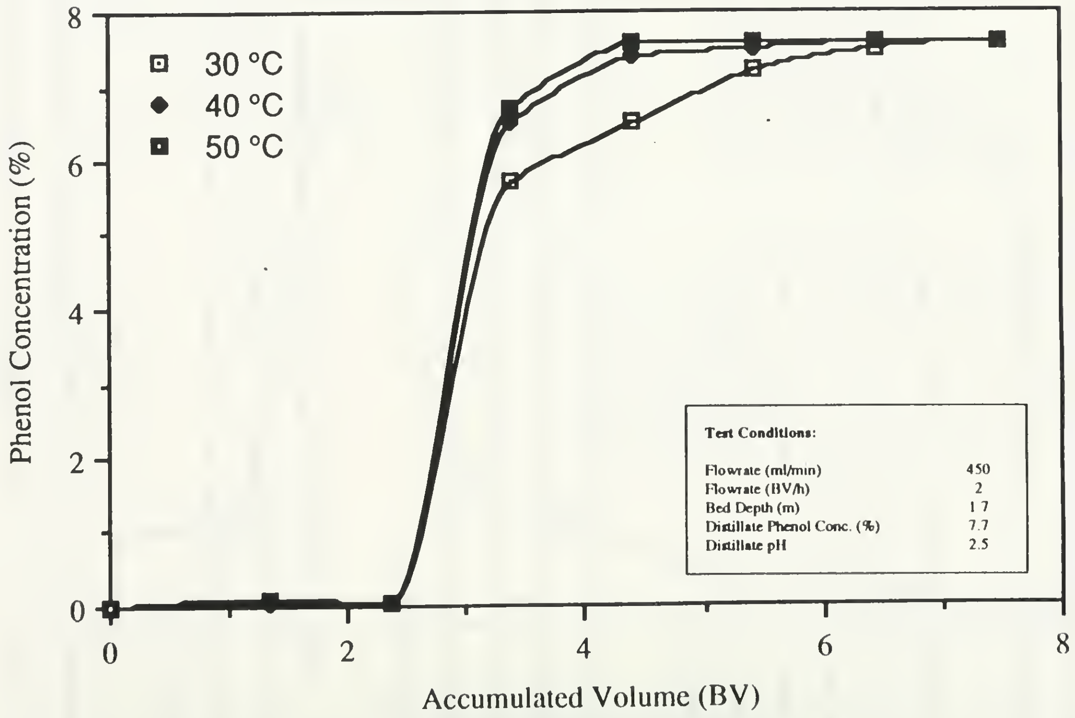
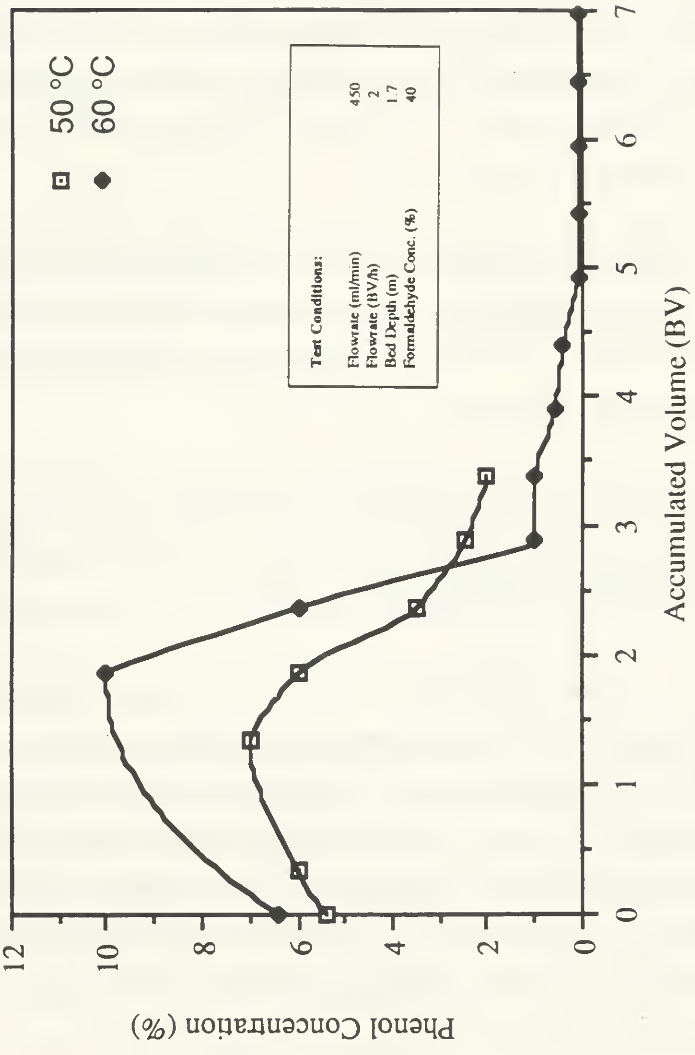


Figure 4.4 - Phenol Elution Profiles at Different Operating Temperatures



In Figure 4.3, phenol breakthrough profiles are shown to become sharper (suggesting a narrower adsorption zone) with increasing operating temperature. This trend is observed mainly because of the higher mass transfer rates that occur at higher operating temperatures. A narrower adsorption zone increases the overall adsorption capacity of the resin bed, although this increase becomes less significant with increasing bed depth. For the 1.7 m bed depth used in the pilot system, adsorption capacity at a 1 g/L phenol breakthrough negligibly increased with a rise in operating temperature from 30°C to 50°C.

A similar trend was observed in Figure 4.4 for the elution profiles, with a sharper profile and corresponding improved elution efficiency occurring with an increase in operating temperature from 50°C to 60°C. The data suggests that the elution process is more sensitive to temperature changes than the loading process.

Based on these results and the fact that formaldehyde is currently stored at the plant at 60°C, operating temperatures of 25 - 30°C for the loading step and 50 - 60°C for the regeneration step were recommended for the phenol recovery process.

4.3 Effect of Hydraulic Loading and Bed Depth on Adsorption Capacity

To properly select a suitable bed depth and hydraulic loading for this application, the minimum bed depth and minimum bed exhaustion rates had to be determined to establish an operating window of possible combinations of these operating parameters. Both hydraulic loading (i.e. superficial liquid velocity through the column) and bed depth define the length of time that the distillate will be in contact with the resin bed. This variable therefore has to be properly optimized (by selecting suitable combinations of hydraulic loading and bed depth) to maximize process performance.

A series of runs were conducted to obtain phenol breakthrough profiles for a range of hydraulic loading and bed depth combinations (Task 2.5, Runs #1 - 5). These concentration profiles are presented in Figures 4.5 to 4.8 covering hydraulic loadings of 2 to 5 m/h and bed depths of 0.6 m to 2.9 m. The distillate phenol concentrations and pH's were 6.2% and 2.9 respectively for hydraulic loadings of 2,3, and 5 m/h, and 7.1% and 3.0 for a hydraulic loading of 4 m/h.

Service times at different bed depths and hydraulic loadings were derived from the profiles in Figures 4.5 to 4.8. This data was then used to construct the bed depth versus service time plots in Figure 4.9. Service time is defined here as the time taken to reach a 1% (w/w) phenol breakthrough concentration in the treated effluent.

A 1% breakthrough concentration was selected after examination of the profiles in Figures 4.5 to 4.8. Particularly in Figures 4.5 and 4.6, the profiles were observed to level off at effluent phenol concentrations between 0.8 and 1% before rising steeply with time. The 1% breakthrough was therefore selected to maximize service time in the loading step while recovering an acceptable amount of phenol.

Linear breakthrough/service time plots suggest that local phenol adsorption rates equal overall adsorption rates, or in other words, that adsorption rates are essentially independent of bed depth on this part of the curve. Figure 4.9 indicates that this is the case only for hydraulic loading up to 4 m/h and bed depths greater than 0.5 m. The y-axis intercept on these plots provides an estimate of minimum bed depth (or width of the adsorption zone) at a particular hydraulic loading. A minimum bed depth of 0.5 m is therefore recommended for the full-scale process. Note that the minimum bed depth defines the minimum contact time at a particular hydraulic loading.

Figure 4.5 - Breakthrough Profiles at 2 m/h Hydraulic Loading

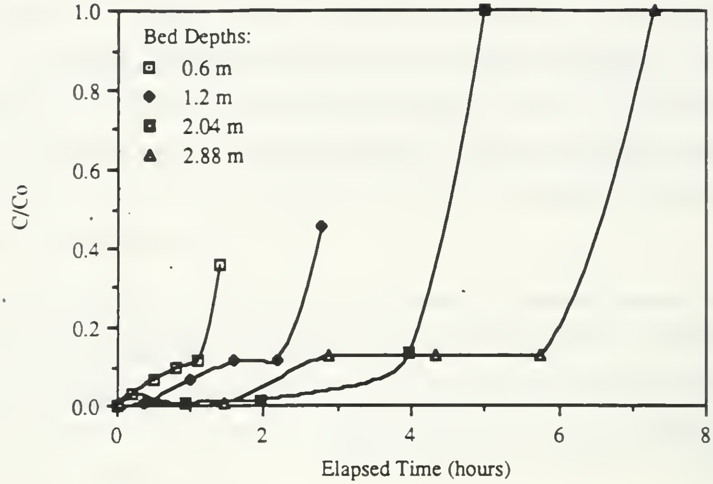


Figure 4.6 - Breakthrough Profiles at 3 m/h Hydraulic Loading

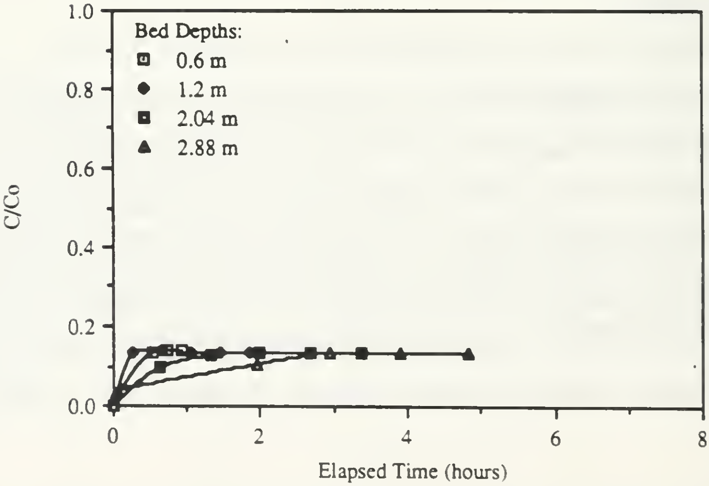


Figure 4.7 - Breakthrough Profiles at 4 m/h Hydraulic Loading

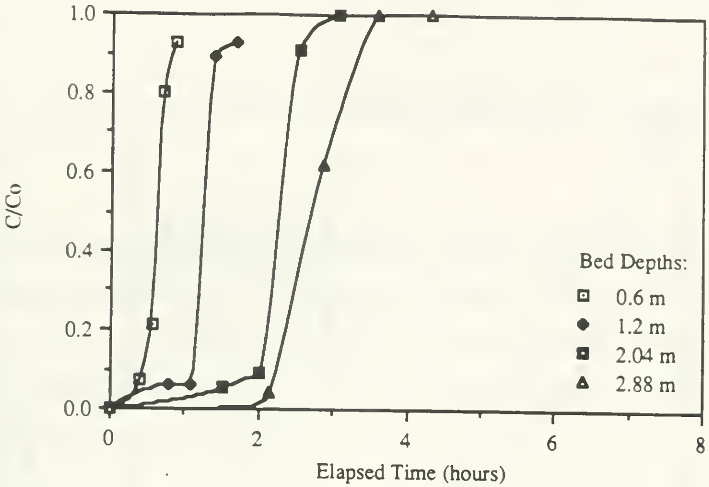


Figure 4.8 - Breakthrough Profiles at 5 m/h Hydraulic Loading

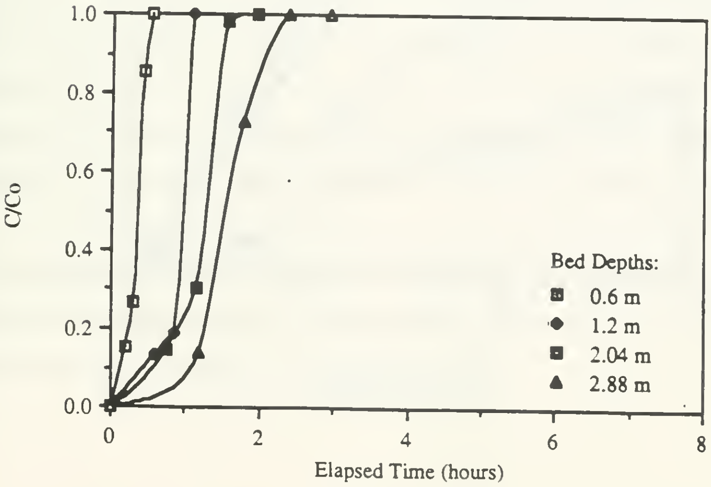
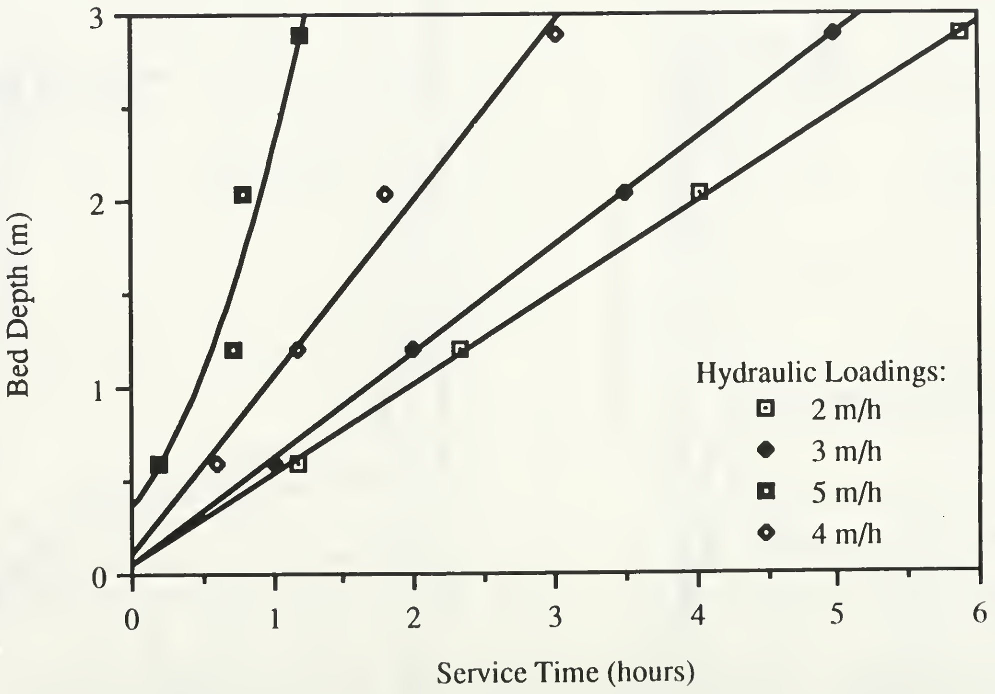


Figure 4.9 - Bed Depth vs Service Time Plots



The operating line plots in Figure 4.10 give another perspective on the range of suitable bed depth/hydraulic loading combinations. Essentially, the objective is to minimize the bed exhaustion rate and therefore operate as close as possible to the horizontal asymptote of the curve. The plots suggest again that for hydraulic loading up to 4 m/h, bed depths of at least 0.6 m should be employed.

4.4 Effect of Hydraulic Loading on Elution Efficiency

Elution profiles were generated at a selected bed depth of 1.2 m and hydraulic loadings of 2, 3, and 4 m/h (Task 2.5, Runs #1-3). The objective of these runs was to determine the optimum hydraulic loading for the elution process. The elution profiles obtained are presented in Figure 4.11 along with relevant test conditions.

The profiles in Figure 4.11 indicate that, for a given volume of eluant, elution efficiency improved with increasing hydraulic loading. The concentration peaks obtained at 2, 3 and 4 m/h were progressively sharper and occurred after less throughput of formaldehyde solution. This trend suggests that hydraulic loading is the predominant operating parameter in the elution process, and consequently that the phenol elution rate is film-diffusion-controlled (i.e. mass transfer through the stagnant boundary layer surrounding the resin particle is the rate limiting step). This is in contrast to the loading process which appeared to be pore-diffusion-controlled and therefore more sensitive to contact time and fairly independent of hydraulic loading (see Section 4.3).

Based on the test results, a hydraulic loading of 4 m/h was selected for longer term runs. Higher hydraulic loadings were not investigated in this study but may have lead to some further improvement in elution efficiency.

Figure 4.10 - Operating Line Plots at Different Hydraulic Loadings

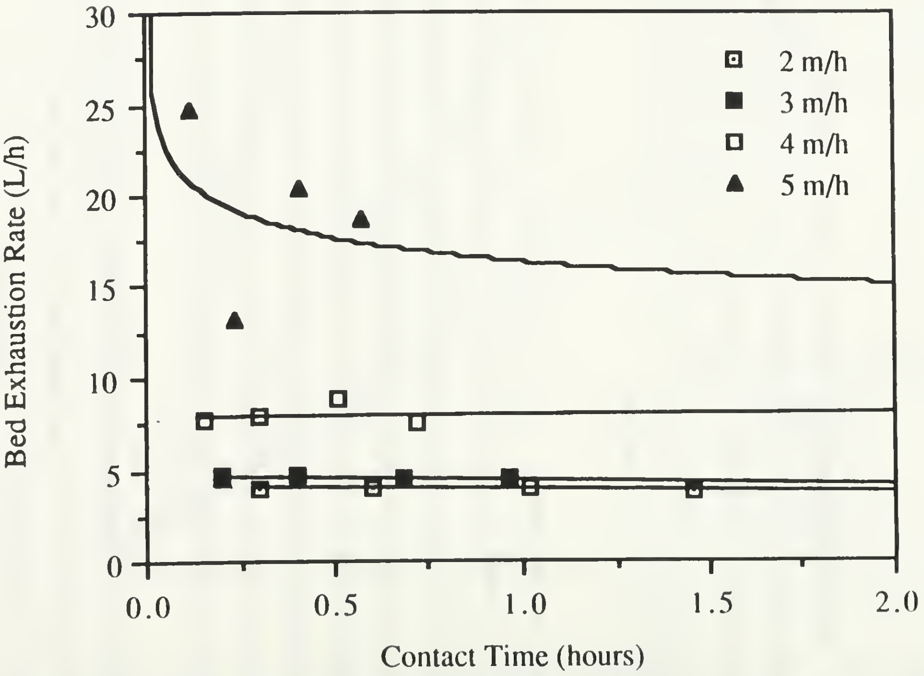
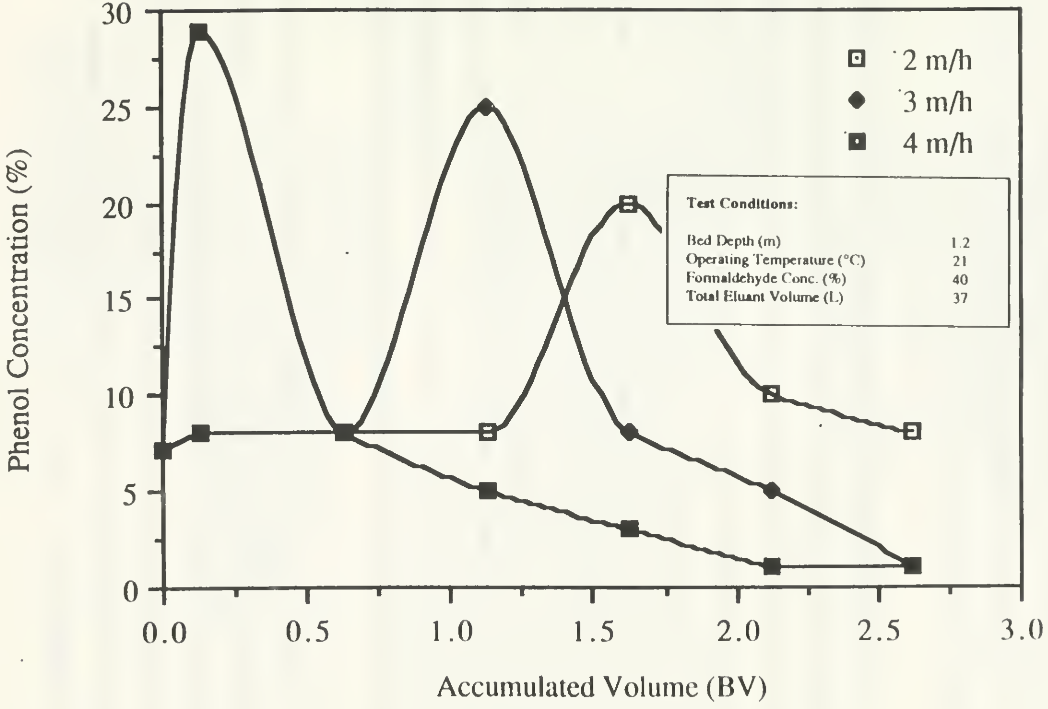


Figure 4.11- Elution Profiles at Different Hydraulic Loadings



4.5 Process Performance in Long Term Tests

A total of 50 loading/regeneration cycles were completed in this phase of the pilot-scale study (Task 3, Runs #1-3). All 50 cycles were carried out using the same resin beds (column #1 and #2). The initial operating conditions were selected based on results obtained in earlier runs and are presented in Table 4.1.

In the first 26 cycles (Task 3, Run #1), adjustments had to be made to the PLC timer settings to optimize process performance. Table 4.2 summarizes the changes that were made and their effect on final effluent concentrations. The objectives of the optimization procedure were to reduce final phenol concentrations leaving the columns below 1% in both the loading and regeneration steps and the final formaldehyde concentration leaving the columns during the rinse step below 1%. By accomplishing this, the composite phenol concentration in the treated distillate stream would remain below 1% (representing at least 86% phenol recovery from a 7% distillate feed), and the adsorption capacity of the resin beds would be reasonably restored after regeneration. Lower effluent phenol concentrations could have been achieved but at the expense of shorter loading times (and consequently lower distillate throughput per cycle) and/or increased usage of formaldehyde solution (and consequently lower phenol/formaldehyde ratios in the eluant stream to be recycled).

The results of cycles 27 to 43 (Task 3, Run #2) and 44 to 50 (Task 3, Run #3) are illustrated in Figure 4.12. Distillate phenol concentrations varied from 3.5 for cycles 27 to 43 to 8.3 for cycles 44 to 50. As a result of this, the duration of the loading step had to be decreased from 120 to 105 minutes to maintain effluent phenol concentrations below 1%. The duration of the regeneration step also had to be increased from 70 to 80 minutes to remove the higher levels of phenol adsorbed on the resin beds.

Table 4.1 - Initial Operating Conditions for Long-Term Tests

Loading Step:

Feed Temperature = 20 - 25°C
Feed Flowrate = 0.39 Lpm
Hydraulic Loading = 3 m/h (1.22 gpm/ft2)
Contact Time = 24 mins (0.4 hrs)
Bed Volume = 9.42 L (Two columns 50% loaded)
Distillate Phenol Concentration = 6.5 - 7.5%
Distillate pH = 2.5 - 3.5

Service Time to 1% Phenol Leakage = 150 mins or 2.5 hrs
Required Distillate Volume = 58.5 L

Regeneration Step:

Feed Temperature = 50°C
Feed Flowrate = 0.52 Lpm
Hydraulic Loading = 4 m/h (1.63 gpm/ft2)
Contact Time = 18 mins (0.3 hrs)
Bed Volume = 9.42 L (Two columns 50% loaded)
Formaldehyde Concentration = 40%

Regeneration Time to 1% Phenol Residual = 60 mins
Required Eluant Volume = 31.2 L

Rinsing Step:

Feed Temperature = >25°C
Feed Flowrate = 1.3 Lpm
Hydraulic Loading = 10 m/h (4.08 gpm/ft2)
Contact Time = 7.2 mins (0.12 hrs)
Bed Volume = 9.42 L (Two columns 50% loaded)

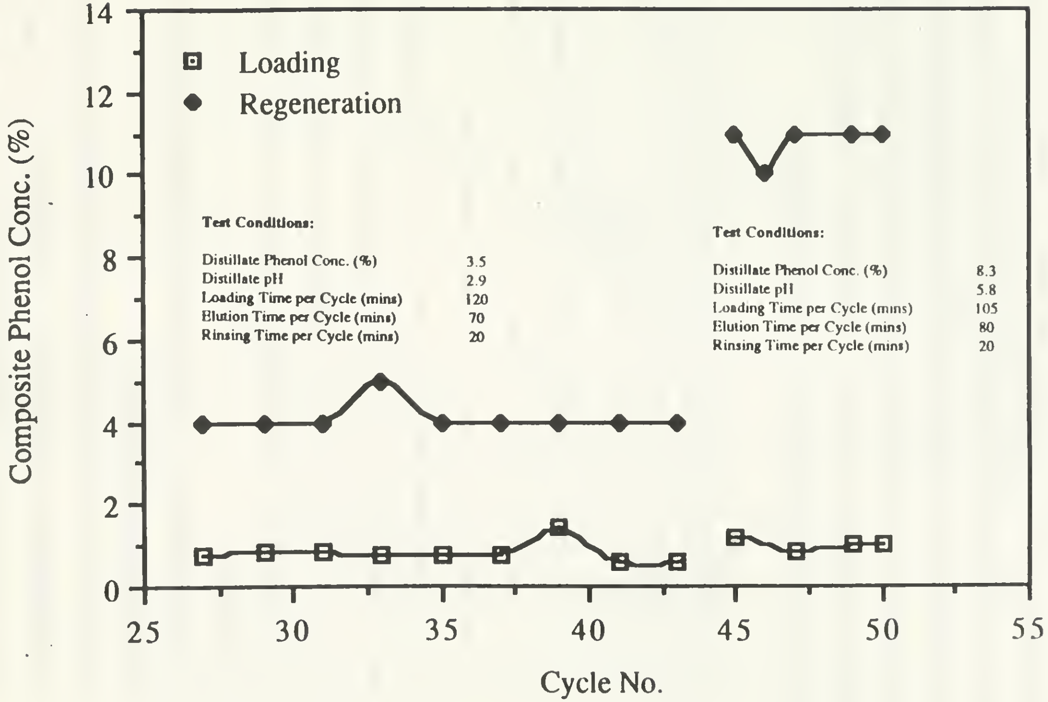
Rinsing Time to 1% Formaldehyde Residual = 15 mins
Required Rinse Volume = 19.5 L

Total Time in Service = 150 mins
Total Time for Regeneration = 75 mins
Total Time on Standby = 75 mins
Total Cycle Time = 300 mins or 5 hrs

Table 4.2 - Optimization of PLC Timer Settings

Cycle No.	Loading Step		Regeneration Step		Rinse Step	
	Step Duration (mins)	Final Phenol Concentration (%)	Step Duration (mins)	Final Phenol Concentration (%)	Step Duration (mins)	Final Formal'de Concentration (%)
1 - 8	150	-	60	-	15	-
9	150	4.9	60	-	15	-
10	140	3.1	60	-	15	-
11	140	-	60	-	5	> 30
11	140	-	60	-	10	15.8
11	140	-	60	-	15	7.3
11	140	-	60	-	20	1.2
12	130	2.5	60	4	20	-
13	120	-	60	-	20	-
14 - 17	120	-	70	-	20	-
18	120	0.95	70	1	20	-
19 - 24	120	-	70	-	20	-
25	120	0.8	70	1	20	-
26	120	0.8	70	1	20	-

Figure 4.12 - Results for Cycles 27 to 50



It should be noted that the composite phenol concentrations reported in Figure 4.12 have not been corrected for the void volume retained in the columns between operating steps. For columns packed 50% (by volume in water) with resin, the void volume was determined to be 68% of the empty column volume or 12.8 L in this case. Phenol concentrations were therefore diluted by up to 1.45 times in the treated distillate stream, and up to about 30% of the collected eluate consisted of untreated distillate feed. The use of columns in the full-scale system with less freeboard or fitted with side ports to remove freeboard volume would result in shorter loading times without necessarily affecting the quality of the treated distillate. Elution and rinse times would also be reduced and eluate phenol concentrations somewhat increased.

The following observations and conclusions were made from the long term tests:

- Phenol concentrations in the treated effluent stream can be maintained at a fairly constant level provided there are no fluctuations in feed conditions.
- Phenol recovery levels ranged from 75% for a distillate phenol concentration of 3.5% to 89% for a concentration of 8.3%. Higher phenol recovery levels are easier to achieve with distillate feeds which contain higher concentrations of phenol.
- Phenol concentrations (and correspondingly the phenol/formaldehyde ratio) in the eluate stream are dependent not only on the volume of formaldehyde used to elute the beds, but also on the phenol concentration in the distillate feed used to load the beds. This is the result of the higher equilibrium adsorption capacities that can be achieved at higher phenol concentrations.
- Phenol concentrations in the eluate stream were concentrated by up to 1.45 times the original distillate phenol concentration.
- A total of 26 L or about 3 bed volumes of fresh water was required to reduce effluent formaldehyde concentrations below 1% after regeneration. The water collected in this rinse step could potentially be used to dilute 50% formaldehyde feed down to 40% for use in the

recovery process. This would eliminate any disposal problems and ensure that all formaldehyde lost in this stream is fully recovered.

- The wide fluctuations observed in distillate concentration and pH had a significant effect on phenol concentrations in both the treated distillate stream and eluate stream for recycle. While fluctuations in eluate phenol concentrations can be tolerated by CanadianOxy, phenol concentrations in the treated distillate stream would have to stay fairly consistent for effective treatment in a subsequent bio-oxidation process. Process efficiency would also be inhibited by a wide range in distillate phenol concentrations considering that timer settings would have to be selected for the worst case. Measures would therefore need to be implemented in a full-scale phenol recovery system to control the effect of variations in distillate phenol concentrations. Possible strategies are either the installation of a distillate equalization tank or a feedback control system involving on-line monitoring of distillate and effluent phenol concentrations. The latter approach could be done using a refractive index method but is likely a more expensive proposition.

5.0 COST ANALYSIS

A preliminary economic analysis was conducted in December 1988 based on bench-scale data for the resin adsorption process. In this analysis, the capital cost for a 5,700 Lpd (1,500 USgpd) phenol recovery system was estimated at $358,000, the annual operating cost at $75,000, and the payback period at 1.8 years when a bio-oxidation unit was included as a secondary treatment process. Annual credits of $110,000 for recovered phenol and $160,000 for eliminating off-site haulage and disposal costs were incorporated into this analysis.

The economics of the phenol recovery process are reassessed here based on operating data generated in the pilot-scale study. Capital and operating cost estimates in this case are believed to be accurate within ±20%. The design basis and specifications used for preparing these estimates are presented in Table 5.1. Note that the column dimensions remain similar to those used in the Phase I cost analysis, but that only two columns are now being used in the full-scale system rather than three. Figure 5.1 contains a possible process flow diagram and mass balance for the full-scale phenol recovery system.

Revised capital and operating cost estimates for a full-scale phenol recovery process were prepared for four different scenarios. Cases #1 and #3 both involve off-site disposal of the distillate as currently practised, but with distillate production rates of 1,600 and 2,100 tonnes/year respectively. CanadianOxy is presently producing distillate at 1,600 tonnes/year but expects this value to increase to 2,100 tonnes/year in the near future with possible plant expansion. In cases #2 and #4, capital and operating costs for a sequencing batch reactor (SBR) have been included for biologically treating distillate effluent leaving the phenol recovery process. This allows credit to be taken for savings in haulage and off-site disposal costs as well as for recovered phenol. Results of the cost analyses are presented in Table 5.2.

Table 5.1 - Design Basis and Specifications for Phenol Recovery System

Design Basis:

Distillate Treatment Rate = 12 Lpm (3.2 gpm)
Distillate Phenol Concentration = 6%
Distillate pH = 3 - 8
Eluant Formaldehyde Concentration = 40%
Required phenol recovery = 90%
Phenol Concentration in Treated Distillate = 0.6%

Operating Temperatures: Loading Step = 25 - 30 °C
Regeneration Step = 50 °C
Rinsing Step = 25 - 30 °C

Operating Period: 8 hours/day, 5 days/week

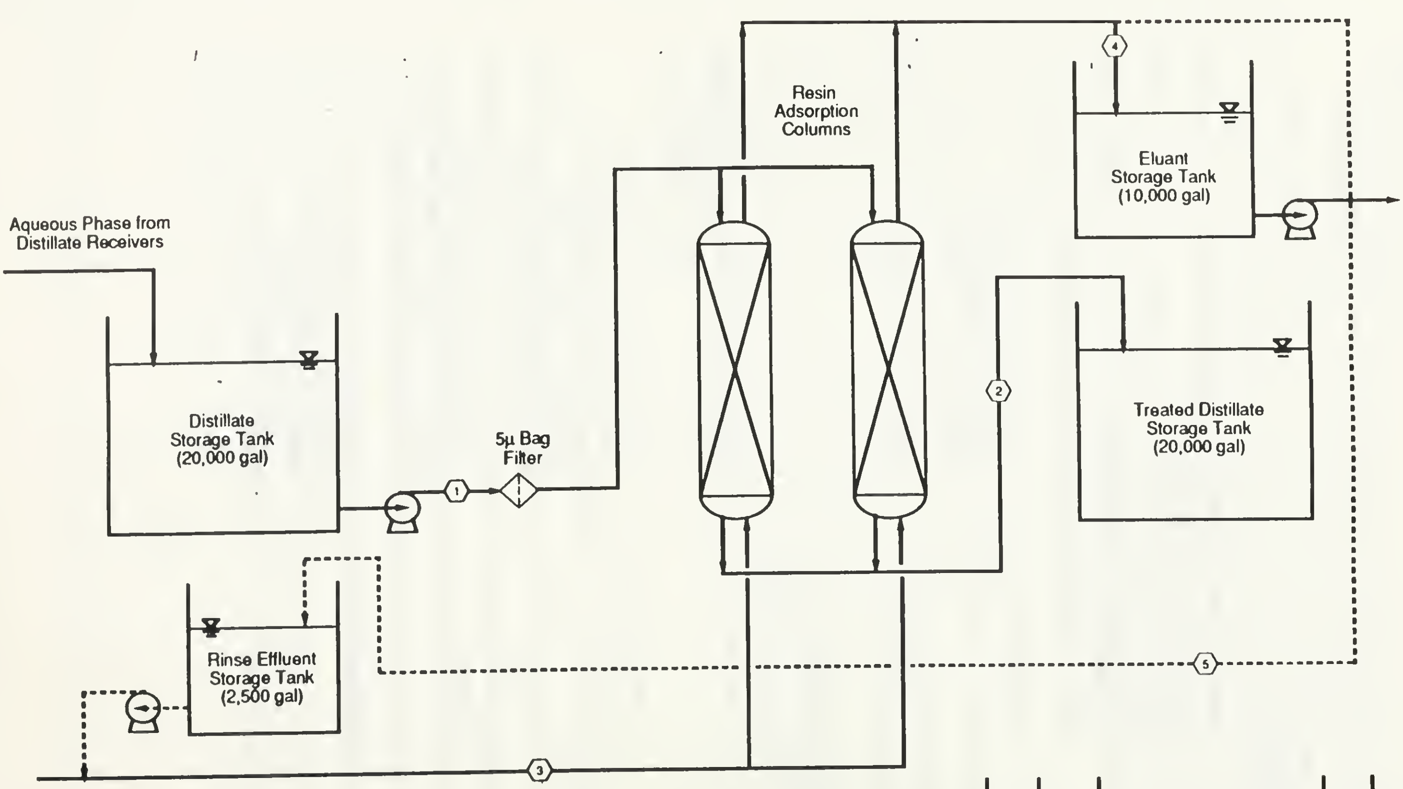
Design Specifications:

Hydraulic Loadings: Loading Step = 3.5 m/h (1.4 gpm/ft2)
Regeneration Step = 4.7 m/h (1.9 gpm/ft2)
Rinsing Step = 11.7 m/h (4.8 gpm/ft2)

Column Dimensions: Column Diameter = 0.51 m (1.7 ft)
Column Depth = 2.4 m (8 ft)
Bed Depth = 1.4 m (4.6 ft)
Bed Volume = 285 L (10 ft3)
Maximum Bed Expansion = 74%

Flowrates: Loading Step = 12 Lpm (3.2 gpm)
Regeneration Step = 16 Lpm (4.2 gpm)
Rinsing Step = 40 Lpm (10.5 gpm)

Step Durations: Loading Step = 110 mins
Regeneration Step = 70 mins
Rinsing Step = 20 mins
Column Standby = 20 mins


Aqueous Phase from Distillate Receivers
Distillate Storage Tank (20,000 gal)
Rinse Effluent Storage Tank (2,500 gal)
5μ Bag Filter
1
Resin Adsorption Columns
2
3
4
5
Eluant Storage Tank (10,000 gal)
Treated Distillate Storage Tank (20,000 gal)

Stream No.	1	2	3	4	6
Stream Description	Distillate Feed	Treated Effluent	Eluant	Eluate	Rinse Effluent
Phenol Conc. (%)	6	0.6	0	10	0
Formaldehyde Conc. (%)	0	0	40	36	20
Design Flowrate (Lpm)	12	12	16	16	40

REVISION #	DATE	DESCRIPTION	BY	APPROVED

SCALE n t s	DRAWN Mario Benito DATE Aug 15 1990	CHK'D R P Carreng DATE Aug 17, 1990	APP'D DATE
CUSTOMER			PROJECT # [illegible]
TITLE	Process Flow Diagram for Resin Adsorption System		DRAWING # Figure 5.1

Table 5.2 - Capital and Operating Costs for Phenol Recovery Process

	Case #1	Case #2	Case #3	Case #4
Basis:				
Annual Distillate Production (tonnes/year)	1600	1600	2100	2100
Distillate Disposal Method after Phenol Recovery	Off-site Disposal	Bio-Treatment	Off-site Disposal	Bio Treatment
Capital Costs:				
Adsorption Columns with Resin (2)	$148,000	$148,000	$175,000	$175,000
Cartridge Filter (10" CPVC Housing)	$600	$600	$600	$600
Process Pumps (3)	$3,000	$3,000	$3,000	$3,000
Treated Distillate Storage Tank (10,000 gal)	$34,400	$34,400	$34,400	$34,400
Eluate Storage Tank (10,000 gal)	$34,400	$34,400	$34,400	$34,400
Rinse Effluent Storage Tank (2,500 gal)	$18,000	$18,000	$18,000	$18,000
Engineering & Installation (25% of Equipment Costs)	$59,600	$59,600	$66,400	$66,400
Royalty on Process Patent (6% of Initial Resin Cost)	$500	$500	$700	$700
Biological Treatment Unit *	-	$150,000	-	$150,000
Total Capital Cost	$299,000	$449,000	$333,000	$483,000
Annual Operating Costs:				
Resin Replacement, every 5 years @ $369/ft3	$1,500	$1,500	$2,000	$2,000
Cartridge Filters @ $4 ea.	$200	$200	$300	$300
Electricity @ 6¢/kWh	$500	$500	$600	$600
Steam	$1,000	$1,000	$1,300	$1,300
Operating Labour (including overhead)	$15,000	$15,000	$15,000	$15,000
Maintenance @ 3% of Direct Capital Costs	$7,200	$7,200	$8,000	$8,000
Depreciation (15 years, Straight-line)	$19,900	$29,900	$22,200	$32,200
Operating and Maintenance of Biological Treatment Unit	-	$50,000	-	$50,000
Sludge Disposal *	-	$5,100	-	$5,100
Total Annual Cost	$45,000	$110,000	$49,000	$115,000
Credits:				
Recovered Phenol @ $1.06/kg	$92,000	$92,000	$120,000	$120,000
Distillate Haulage and Disposal @ $73.4/tonne	-	$117,000	-	$117,000
Distillate Haulage and Disposal @ $319/tonne **	-	-	-	$160,000
Net Annual Cost	($47,000)	($99,000)	($71,000)	($282,000)
Payback Period (years)	6.4	4.5	4.7	1.7

* *Costs supplied by CanadianOxy.*

** *Cost for incremental amounts of distillate over 1,600 tonnes/year.*

In determining capital costs, a budgetary quote for a custom-designed, counter-currently regenerated, two column system was obtained from IWT-Himsley. The system would be fitted with a PLC, pneumatic activated valves and all CPVC piping and fittings. Cost estimates were also solicited from suppliers for process pumps, a cartridge filter and jacketted SS304 L storage tanks. Note that the three SS304 L tanks required for the process account for about 36% of the total direct capital costs in cases #1.

The following assumptions were made in estimating operating costs:

- System operation for 8 hours/day, 5 days/week and 50 weeks/year
- An operating life of 15 years (compared to 10 years assumed in the Phase I cost analysis)
- Resin replacement required once every 5 years
- Average operating labour requirements of 2 hours per day. For an annual full-time salary of $60,000 (including overhead), this would amount to $15,000 annually.
- Maintenance costs amount to 3% of direct capital costs
- Depreciation costs are calculated by the straight-line method
- 90% of distillate phenol is recovered

Payback periods in Table 5.2 are shown to range from 1.7 to 6.4 years depending on the basis for the cost analysis. In case #2, a payback period of 4.5 years was estimated using the same basis as that used for the Phase I economic assessment. The increase in payback period from 1.8 years (obtained in the Phase I study) to 4.5 years is attributed primarily to the following reasons:

- No storage tank requirements were included in the Phase I capital cost esimtate. The cost for three jacketted, SS304 L tanks are quite substantial, accounting for 36% of direct capital costs for handling 1,600 tonnes of distillate per year and 33% for handling 2,100 tonnes of distillate per year.

- The estimated annual operating cost increased by 47% in this analysis compared to that estimated in the Phase I study. The increase was mainly due to the inclusion of sludge disposal costs and to significantly higher operating and maintenance cost estimates for the SBR biological treatment unit.
- Credits taken for recovered phenol and savings in off-site distillate disposal costs were about 23% lower in this analysis than in the Phase I study. The phenol recovery credit has decreased because of a drop in phenol prices over the last two years. A lower haulage and disposal cost for distillate was also incorporated into this assessment to reflect current costs to CanadianOxy.

The cost data presented in Table 5.2 are felt to represent a fairly realistic picture of the cost and economic benefits of recovering phenol at the CanadianOxy plant. In fact, the estimates are likely to be on the conservative side considering that phenol prices have started to increase again, distillate disposal costs will continue to increase and the cost of the SS304 L storage tanks can potentially be reduced by decreasing tankage capacity or sourcing used tanks. The payback periods for the four cases clearly indicate that a biological treatment plant should accompany the installation of a recovery system in order to realize attractive returns on capital investment. The data also suggests that any future expansion of CanadianOxy's operations will contribute significantly to improving the economic attractiveness of the recovery process. A payback period of only 1.7 years was estimated for treating 2,100 tonnes/year of distillate with resin adsorption followed by biological oxidation.

REFERENCES

1. APHA, AWWA and WPCF, "Standard Methods for the Examination of Water and Wastewater", 17th ed., APHA, AWWA and WPCF, Washington, D.C., 1989.

2. Weber, W.J., "Physicochemical Processes for Water Quality Control", Wiley-Interscience, New York, N.Y., 1972.

APPENDIX A

Pilot Study Workplan

REVISED WORKPLAN FOR TASKS 2 AND 3

Task 2 - Column Testing and Process Optimization

Task 2.1 - Verification of Bench-Scale Results

Objective: To verify the operating capacities and elution efficiencies observed for the XAD7 resin in bench-scale column tests.

Procedure:

a) Pilot unit should be put into manual mode by turning the mode selector switch on the lower left corner of the control panel to the HAND position.

b) With solenoid valves SV1, SV4 and SV7 activated, switch on P1 to feed distillate through columns #1 and #2. Set distillate flowrate at 0.44 Lpm (2 BV/h) quickly by adjusting valves V1 and V2. Distillate temperature should be between 20 and 25 °C.

c) Effluent samples should then be taken after every bed volume or every 30 minutes until at least 8 bed volumes are collected.

d) At completion of loading step, turn pump P1 off and deactivate SV1, SV4 and SV7.

e) Replumb columns #1 and #2 to permit co-current regeneration.

f) Activate SV3, SV5 and SV6 before switching on pump P2 to feed eluant co-currently though columns #1 and #2. Set eluant flowrate at 0.44 Lpm (2 BV/h) quickly by adjusting valves V3 and V4. The eluant should be 40% formaldehyde and introduced at a temperature between 20 and 25 °C.

g) Sample column effluent every 0.5 bed volumes or 15 minutes until a minimum of 4 bed volumes are collected.

h) At completion of elution step, rinse beds with warm tap water by momentarily turning pump P2 off, deactivating SV3 and activating SV2 before switching P2 back on. Continue rinse step for a minimum of 10 minutes at 10 BV/h (2.2 Lpm).

i) Shut off unit after rinsing is complete allowing beds to remain immersed in water.

Task 2.2 - Evaluate Counter-Current verses Co-Current Elution

Objective: To evaluate the degree of increased efficiency obtained with counter-current as opposed to co-current elution.

Procedure: a) Repeat task 2.1 procedure after replumbing columns #1 and #2 for counter-current regeneration.

Task 2.3 - Investigate Effect of Distillate Temperature on Bed Capacity

Objective: To construct breakthrough profiles for distillate feeds of different temperatures in order to evaluate the effect of temperature on the extent of phenol adsorption. Diffusion rates generally increase at higher temperatures resulting in more phenol adsorption onto the resin bed. The data will be useful for deciding on a trade-off between the increased phenol uptake at higher temperatures and the higher operating costs associated with maintaining the feed at elevated temperatures.

Procedure:

a) Follow task 2.1 procedure except as otherwise noted.

b) Pilot unit should be plumbed for counter-current regeneration.

c) Repeat procedure for distillate temperatures of 30, 40 and 50 °C.

d) After each loading step, the beds should be eluted counter-currently with 40% formaldehyde at 2 BV/h (0.44 Lpm) for exactly 3 hours and rinsed with warm tap water at 10 BV/h (2.2 Lpm) for 10 minutes before beginning the next loading step.

e) The eluate stream for each regeneration step should be collected in a separate container so that a composite sample of the stream can be taken for phenol analysis.

f) In the loading step, the column effluent should be sampled at the intervals suggested in Task 2.1, step (c).

Note: Both feed and effluent temperatures should be monitored during the loading step so that the average operating temperature in the beds can be estimated.

Task 2.4 - Investigate Effect of Eluant Temperature on Elution Efficiency

Objective: To construct elution profiles for eluant feeds at different temperatures in order to evaluate the effect of temperature on the efficiency with which phenol is removed from the resin beds. Again, because of the higher diffusion rates that occur at higher temperatures, the beds are expected to be eluted more efficiently as the eluant temperature is increased. Advantage can thus be taken of the fact that formaldehyde is stored at about 60 °C.

Procedure:

a) Follow task 2.1 procedure except as otherwise noted.

b) Pilot unit should be plumbed for counter-current regeneration.

c) Before each elution step, load beds for two hours at a distillate flowrate of 5 BV/h (1.1 Lpm).

d) Repeat procedure for eluant temperatures of 50 and 60 °C.

e) After the elution step, the beds should be rinsed with warm tap water at 10 BV/h (2.2 Lpm) for 10 minutes before beginning the next loading step.

f) The effluent stream for each loading step should be collected in a separate container so that a composite sample of the stream can be taken for phenol analysis.

g) In the regeneration step, the eluate stream should be sampled at the intervals suggested in Task 2.1, step (g).

Note: Both feed and effluent temperatures should be monitored during the elution step so that the average operating temperature in the beds can be estimated.

Task 2.5 - Investigate Effect of Hydraulic Loading and Bed Depth on Bed Capacity

Objective: To generate breakthrough profiles for different hydraulic loadings at varying bed depths. The data gathered in these tests will be used to construct plots of bed depth versus service time and bed exhaustion rate versus contact time. An optimum bed depth and hydraulic loading for the loading step will be determined from these plots.

Procedure:

a) Follow task 2.1 procedure except as otherwise noted.

b) The pilot unit should be replumbed to permit all four columns to be operated in series with counter-current regeneration. Columns #1 and #2 should only be filled with resin to 50 % of their volume and should receive distillate feed before columns #3 and #4, each filled to 70% of their volume. Cumulative bed depths are therefore 60, 120, 204 and 288 cm after columns #1, #2, #3 and #4 respectively.

c) Feed distillate to the columns at hydraulic loadings of 2, 3 and 5 m/h. Refer to Table 1 for corresponding flowrates through columns in Lpm.

d) In the loading step, solenoid valves SV1, SV7 and SV8 have to be activated, and pump P1 turned on.

e) At each distillate flowrate, sample effluent from the bottoms of column #1, #2, #3 and #4 consecutively after every bed volume for a total of six bed volumes. Note that bed depths increase after each column, and therefore the time interval between sampling will be different for each column at a particular flowrate. Required sampling frequencies are given in Table 1 as a function of bed depth and volumetric flowrate.

f) After each loading step, the beds should be eluted counter-currently with 40% formaldehyde at 2 BV/h (0.75 Lpm) for exactly 3 hours and rinsed with warm tap water at 5 BV/h (1.9 Lpm) for 20 minutes before beginning the next loading step.

g) In the elution step, solenoid valves SV3, SV6 and SV9 have to be activated, and pump P2 turned on. Pump P2 should be turned off, and solenoid valves SV2, SV6 and SV9 activated to carry out the rinsing step.

h) The eluate stream for each elution step should be collected in a separate container so that a composite sample of the stream can be taken for phenol analysis.

Note: If possible , all task 2.5 tests should be carried out with the same distillate feed.

Task 2.6 - Investigate Effect of Hydraulic Loading on Elution Efficiency

Objective: To generate elution profiles for different hydraulic loadings at a selected bed depth. The elution profiles will be used to determine the optimum hydraulic loading for the elution step. A bed depth will be selected based on the results of Task 2.5.

Procedure:

a) Follow task 2.1 procedure except as otherwise noted.

b) The pilot unit should be replumbed to permit the required number of columns to be operated in series with counter-current regeneration.

c) Before each elution step, load beds for two hours at a distillate flowrate of 5 BV/h.

d) The effluent stream for each loading step should be collected in a separate container so that a composite sample of the stream can be taken for phenol analysis.

e) In the loading step, solenoid valves SV1, SV7 and SV8 have to be activated, and pump P1 turned on.

f) The elution step should be carried out with eluant hydraulic loadings of 1, 2 and 3 m/h. Refer to Table 1 for corresponding flowrates through columns in Lpm.

g) In the elution step, solenoid valves SV3, SV6 and SV9 have to be activated, and pump P2 turned on.

h) At each eluant flowrate, sample column effluent after every half a bed volume for a total of 4 bed volumes.

i) After the elution step, the beds should be rinsed with warm tap water at 10 BV/h for 10 minutes before beginning the next loading step.

j) Pump P2 should be turned off, and solenoid valves SV2, SV6 and SV9 activated to carry out the rinsing step.

Note: If possible , all task 2.5 tests should be carried out with the same distillate feed.

Task 3 - Long-term Column Operation

Objective: To conduct a long-term evaluation of the phenol recovery process at the optimum operating conditions established in Task 2. In particular, the following concerns will be investigated:

i) Decline in resin performance due to chemical poisoning, physical deterioration and/or mechanical attrition.

ii) The ability of the process to adequately recover phenol from distillate feeds of varying phenol concentration and pH.

iii) Minimization of eluant requirements to permit maximum phenol concentrations in the raffinate for recycle to reactors.

iv) Shortening of rinse step to minimize production of formaldehyde-contaminated wastewater.

v). Production of sufficient raffinate for CanadianOxy personnel to determine impact of recycled raffinate on their final product.

Procedure:

a) All four columns should be charged with XAD7 resin to 50% of their empty volumes and plumbed to permit simultaneous loading and regeneration of columns #1 and #2, and columns #3 and #4 with counter-current flow (i.e return pilot plant to its original flow configuration).

b) Set Step 0 to Step 5 counters at predetermined values before beginning run. (These values will be defined once Task 2 tests have been completed.) The procedure for setting the counters with the timer/counter access module is given in the appendix.

c) With all toggle switches in the OFF position, pilot unit should be put into automatic mode by turning the mode selector switch on the lower left corner of the control panel to the AUTO position.

d) Run the process under an optimum set of operating conditions as determined in Task 2 (i.e. optimum distillate temperature, eluant temperature, hydraulic loading for loading step, hydraulic loading for elution step and bed depth).

e) Composite samples of the raffinate and treated distillate should be taken after each cycle until steady-state conditions are reasonably approached and after every third cycle thereafter.

f) Adjustments can be made to the counter settings during the run as required to produce acceptable effluent and raffinate streams.

g) A minimum of 50 service cycles should be conducted with each bed before terminating the run.

Table 1 - Flowrate Conversions at Different Bed Depths

Bed Depth	Bed Volume	Hydraulic Loading		Volumetric Flowrate			Sampling Frequency
(cm)	(L)	(m/h)	(gpm/ft2)	(BV/h)	(Lpm)	(gpm)	(mins.)
60	4.71	1	0.41	1.67	0.13	0.03	36
60	4.71	2	0.82	3.33	0.26	0.07	18
60	4.71	3	1.22	5.00	0.39	0.10	12
60	4.71	4	1.63	6.67	0.52	0.14	9
60	4.71	5	2.04	8.33	0.65	0.17	7
120	9.42	1	0.41	0.83	0.13	0.03	72
120	9.42	2	0.82	1.67	0.26	0.07	36
120	9.42	3	1.22	2.50	0.39	0.10	24
120	9.42	4	1.63	3.33	0.52	0.14	18
120	9.42	5	2.04	4.17	0.65	0.17	14
168	13.19	1	0.41	0.60	0.13	0.03	101
168	13.19	2	0.82	1.19	0.26	0.07	50
168	13.19	3	1.22	1.79	0.39	0.10	34
168	13.19	4	1.63	2.38	0.52	0.14	25
168	13.19	5	2.04	2.98	0.65	0.17	20
204	16.02	1	0.41	0.49	0.13	0.03	122
204	16.02	2	0.82	0.96	0.26	0.07	61
204	16.02	3	1.22	1.47	0.39	0.10	41
204	16.02	4	1.63	1.96	0.52	0.14	31
204	16.02	5	2.04	2.45	0.65	0.17	24
288	22.62	1	0.41	0.35	0.13	0.03	173
288	22.62	2	0.82	0.69	0.26	0.07	86
288	22.62	3	1.22	1.04	0.39	0.10	58
288	22.62	4	1.63	1.39	0.52	0.14	43
288	22.62	5	2.04	1.74	0.65	0.17	35

APPENDIX B

Operating Log Sheets

LOG SHEET

Date: Jan. 10, 1990 **Task 2.1 - Run #:** 1 **Operator:** Tony Moxam

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
3:15	0	6	15	0
3:45	0.5	6	15	-
4:15	1	6	15	5.4 *
4:45	1.5	6	15	0.25
8:30	1.5	6	15	0.25
9:00	2	6	15	0.25
9:30	2.5	6	15	0.15
10:00	3	6	15	4.9
10:30	3.5	6	15	5.1
11:00	4	6	15	5.2
11:30	4.5	6	15	5.1
12:00	5	6	15	5.4

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
2:00	0	3	30	0
2:15	0.25	3	30	7
2:30	0.5	3	30	6
2:45	0.75	5	30	-
3:00	1	5	30	7
3:15	1.25	5	30	6
3:30	1.5	5	30	3.5
3:45	1.75	5	30	2.5
4:00	2	5	30	2

Operating Conditions:

Feed Temperature = 15°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Distillate Feed Conditions:

Phenol Concentration = 5.4%

pH = 3.3

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH =

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 70% by volume.
- No rinse step carried out before loading.
- Loading step stopped at 4:45, Jan. 10 and continued at 8:30, Jan. 11.
- Regeneration step started 2 hours after loading step was completed.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Jan. 23, 1990

Task 2.3 - Run #: 1

Operator: Tony Moxam

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
9:25	0	5	25	0
10:00	0.5	5	25	0.3 *
10:30	1	5	29	0.1
11:00	1.5	5	28	0.05
11:30	2	5	27	5.7
12:00	2.5	5	27	6.5
12:30	3	5	27	7.2
13:00	3.5	5	27	7.5
13:25	4	5	28	7.6

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
13:35	0	6	26	-
16:45	0 - 3	6	32	6

Operating Conditions:

Feed Temperature = 30°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Distillate Feed Conditions:

Phenol Concentration = 7.8%

pH = 2.6

Operating Conditions:

Feed Temperature = 45°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH =

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 70% by volume.
- Regeneration step started 24 hours after completion of loading step.
- A composite sample of the effluent was taken over the 3 hour regeneration step.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Jan. 26, 1990

Task 2.3 - Run #: 2

Operator: Tony Moxam

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
9:00	0	5	22	0
9:30	0.5	5	29	0.1 *
10:00	1	5	31	0.05
10:30	1.5	5	30	0.05
11:00	2	5	30	6.5
11:30	2.5	5	30	7.4
12:00	3	5	30	7.5
12:30	3.5	5	30	7.6
12:55	4	5	29	7.6

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
13:25	0	2	28	-
16:25	3	9	31	6

Operating Conditions:

Feed Temperature = 40°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Distillate Feed Conditions:

Phenol Concentration = 7.7%

pH = 2.5

Operating Conditions:

Feed Temperature = 49°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH =

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 70% by volume.
- A composite sample of the effluent was taken over the 3 hour regeneration step.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Feb. 15, 1990

Task 2.3 - Run #: 3

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
9:00	0	6	22	0
9.30	0.5	6	22	0.3 *
10:00	1	6	31	0.1
10:30	1.5	6	34	0.05
11:00	2	6	33	6.7
11:30	2.5	6	34	7.6
12:00	3	6	34	7.6
12:30	3.5	6	36	7.6
13:00	4	6	34	7.6

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
13:15	0	1	31	
16:15	3	4	32	8

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Distillate Feed Conditions:

Phenol Concentration = 7.7%

pH = 2.5

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH =

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 70% by volume.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- Problems experienced in keeping pressure drop across bed in downflow at acceptable levels.

* *These phenol concentrations were neglected due to possible sample contamination*

LOG SHEET

Date: Feb. 21, 1990

Task 2.4 - Run #: 1

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
9:30	0	10	26	-
12:30	3	12	27	-

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
12:30	0	2	26	-
13:15	0.75	2	26	4 *
13:30	1	2	28	4 *
13:45	1.25	2	32	10
14:00	1.5	2	34	6
14:15	1.75	2	35	1
14:30	2	2	35	1
14:45	2.25	2	36	0.6
15:00	2.5	2	35	0.4
15:15	2.75	2	35	0.1
15:30	3	2	35	0.1
15:45	3.25	2	35	0.1
16:00	3.5	2	35	0
16:15	3.75	2	35	0

Operating Conditions:

Feed Temperature = 32°C

Feed Flowrate = 1.1 Lpm

Bed Volume = 13.2 L

Distillate Feed Conditions:

Phenol Concentration = 6.4%

pH =

Operating Conditions:

Feed Temperature = 60°C

Feed Flowrate = 0.45 Lpm

Bed Volume = 13.2 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH =

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 70% by volume.
- A composite sample of the effluent was taken over the 3 hour loading step.
- Low pressure switch tripped at 13:15.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 4, 1990

Task 2.5 - Run #: 1.1

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.3	-	-	0.05 *
-	0.6	-	-	0.18
-	0.9	-	24	0.43
-	1.2	-	24	0.6
-	1.5	-	24	0.7
-	1.8	-	24	2.2

Operating Conditions:

Feed Temperature = 20.5°C

Feed Flowrate = 0.26 Lpm

Bed Volume = 4.7 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 4.7 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps, observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #1 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Very top of resin bed had a rust colour at end of loading step, and distillate above bed was cloudy.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 4, 1990

Task 2.5 - Run #: 1.2

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.6	-	-	0.18 *
-	1.2	-	-	0.05
-	1.8	-	24	0.4
-	2.4	-	24	0.7
-	3	-	24	0.73
-	3.6	-	24	2.8

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 20.5°C

Feed Flowrate = 0.26 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9.

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #2 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Some cloudiness observed in distillate above bed.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 4, 1990 **Task 2.5 - Run #:** 1.3 **Operator:** Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	1.02	-	-	<0.05 *
-	2.03	-	-	<0.05
-	3.05	-	24	0.65
-	4.07	-	24	0.75
-	5.08	-	24	0.83
-	6.1	-	24	6.2

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 20.5°C

Feed Flowrate = 0.26 Lpm

Bed Volume = 16 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 16 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #3 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 4, 1990

Task 2.5 - Run #: 1.4

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	1.4	-	-	<0.05 *
-	2.9	-	-	0.05
-	4.3	-	24	0.79
-	5.7	-	24	0.79
-	7.2	-	24	0.79
-	8.7	-	24	6.5

Operating Conditions:

Feed Temperature = 20.5°C

Feed Flowrate = 0.26 Lpm

Bed Volume = 22.6 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 22.6 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #4 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 9, 1990

Task 2.5 - Run #: 2.1

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.2	-	-	0.96 *
-	0.4	-	-	1.15 *
-	0.6	-	24	0.96 *
-	0.8	-	24	0.83
-	1	-	24	0.88
-	1.2	-	24	0.88

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	4

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 4.7 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 4.7 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #1 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Very top of resin bed had a rust colour at end of loading step, and distillate above bed was cloudy.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 9, 1990

Task 2.5 - Run #: 2.2

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.4	-	-	0.7
-	0.8	-	-	0.83
-	1.2	-	-	0.88
-	1.6	-	-	0.83
-	2	-	-	0.83
-	2.4	-	-	0.83

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	4

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #2 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Distillate was observed to be cloudy between top of column and resin bed.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 9, 1990 **Task 2.5 - Run #:** 2.3 **Operator:** Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.68	-	-	0.4
-	1.37	-	-	0.6
-	2.05	-	-	0.79
-	2.73	-	-	0.83
-	3.42	-	-	0.83
-	4.1	-	-	0.83

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	4

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 16.0 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 16.0 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #3 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 9, 1990

Task 2.5 - Run #: 2.4

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.97	-	-	0.18
-	1.93	-	-	0.43
-	2.9	-	-	0.65
-	3.87	-	-	0.83
-	4.83	-	-	0.83
-	5.77	-	-	0.83

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	4

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 22.6 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 22.6 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #4 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 11, 1990

Task 2.5 - Run #: 3.1

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.12	-	-	<0.05
-	0.23	-	-	7.9 *
-	0.35	-	-	0.96
-	0.47	-	-	1.65
-	0.58	-	-	5.3
-	0.7	-	-	6.3

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.65 Lpm

Bed Volume = 4.7 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 4.7 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #1 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Very top of resin bed had a rust colour at end of loading step, and distillate above bed was cloudy.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 11, 1990

Task 2.5 - Run #: 3.2

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.23	-	-	5.8 *
-	0.47	-	-	8.2 *
-	0.7	-	-	0.96 *
-	0.93	-	-	0.83
-	1.17	-	-	1.15
-	1.4	-	-	6.2

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.65 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #2 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- The distillate between the top of the column and the resin bed was observed to be cloudy.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 11, 1990

Task 2.5 - Run #: 3.3

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.4	-	-	3.0 *
-	0.8	-	-	4.2 *
-	1.2	-	-	0.92
-	1.6	-	-	1.89
-	2	-	-	6.1
-	2.4	-	-	6.6

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.65 Lpm

Bed Volume = 16.0 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 16.0 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #3 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 11, 1990

Task 2.5 - Run #: 3.4

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.58	-	-	1.86 *
-	1.16	-	-	1.20 *
-	1.75	-	-	0.88
-	2.33	-	-	4.5
-	2.92	-	-	6.5
-	3.5	-	-	6.5

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	25	0
-	3	-	28	5

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.65 Lpm

Bed Volume = 22.6 L

Distillate Feed Conditions:

Phenol Concentration = 6.2%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 22.6 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #4 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 23, 1990

Task 2.5 - Run #: 4.1

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.3	5	20	1.54 *
-	0.45	5	20	0.48 *
-	0.6	5	20	0.32
-	0.75	5	20	0.96
-	0.9	5	20	3.6
-	1.05	5	20	4.2

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	6

Operating Conditions:

Feed Temperature = 25°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 4.7 L

Distillate Feed Conditions:

Phenol Concentration = 4.5%

pH = 2.9

Operating Conditions:

Feed Temperature = 55°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 4.7 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #1 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Distillate not as cloudy as in last three stes of runs.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 23, 1990 **Task 2.5 - Run #:** 4.2 **Operator:** Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.6	5	20	2.1 *
-	0.9	5	20	0.38 *
-	1.2	5	20	0.28
-	1.5	5	20	0.28
-	1.8	5	20	4
-	2.1	5	21	4.2

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	6

Operating Conditions:

Feed Temperature = 25°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 4.5%

pH = 2.9

Operating Conditions:

Feed Temperature = 55°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #2 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Distillate not as cloudy as in last three sets of runs.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 23, 1990

Task 2.5 - Run #: 4.3

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	1.03	5	20	1.4 *
-	1.55	5	20	0.28
-	2.07	5	21	0.24
-	2.58	5	21	0.42
-	3.1	5	22	4.1
-	3.62	5	22	4.5

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	6

Operating Conditions:

Feed Temperature = 25°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 16.0 L

Distillate Feed Conditions:

Phenol Concentration = 4.5%

pH = 2.9

Operating Conditions:

Feed Temperature = 55°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 16.0 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #3 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Distillate not as cloudy as in last three stes of runs.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 23, 1990 **Task 2.5 - Run #:** 4.4 **Operator:** Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	1.43	5	20	1.22 *
-	2.15	5	21	0.18
-	2.86	5	21	0.18
-	3.58	5	22	2.8
-	4.3	5	22	4.5
-	5.02	5	22	4.5

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	6

Operating Conditions:

Feed Temperature = 25°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 22.6 L

Distillate Feed Conditions:

Phenol Concentration = 4.5%

pH = 2.9

Operating Conditions:

Feed Temperature = 55°C

Feed Flowrate = 0.75 Lpm

Bed Volume = 22.6 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #4 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Distillate not as cloudy as in last three stes of runs.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 30, 1990 **Task 2.5 - Run #:** 5.1 **Operator:** Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.3	5	20	6.5 *
-	0.45	5	20	1.82
-	0.6	5	20	0.32
-	0.75	5	20	0.26
-	0.9	5	20	4.5
-	1.05	5	20	6.7

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	5

Operating Conditions:

Feed Temperature = 22°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 4.7 L

Distillate Feed Conditions:

Phenol Concentration = 7.1%

pH = 3.0

Operating Conditions:

Feed Temperature =

Feed Flowrate = 0.75 Lpm

Bed Volume = 4.7 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #1 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- Top of columns #1 and 2 were both fairly cloudy.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 30, 1990

Task 2.5 - Run #: 5.2

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.6	5	20	2.3 *
-	0.9	5	20	0.82 *
-	1.2	5	20	0.18
-	1.5	5	20	0.26
-	1.8	5	20	5.3
-	2.1	5	21	6.3

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	5

Operating Conditions:

Feed Temperature = 22°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 7.1%

pH = 3.0

Operating Conditions:

Feed Temperature =

Feed Flowrate = 0.75 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #2 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- After 2.75 hours of loading, top of column #1 had a rust colour band about 2" thick with streaks halfway down the column.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 30, 1990

Task 2.5 - Run #: 5.3

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	1.03	5	20	2.7 *
-	1.55	5	20	0.28 *
-	2.07	5	21	0.14
-	2.58	5	21	2.2
-	3.1	5	22	6.4
-	3.62	5	22	6.5

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	5

Operating Conditions:

Feed Temperature = 22°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 16.0 L

Distillate Feed Conditions:

Phenol Concentration = 7.1%

pH = 3.0

Operating Conditions:

Feed Temperature =

Feed Flowrate = 0.75 Lpm

Bed Volume = 16.0 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #3 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- After 2.75 hours of loading, top of column #4 had a rust colour band about 1/2" thick, but nothing was noticed in column #3.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Apr. 30, 1990

Task 2.5 - Run #: 5.4

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	1.43	5	20	2.1 *
-	2.15	5	21	0.32 *
-	2.86	5	21	0.2
-	3.58	5	22	0.14
-	4.3	5	22	5.1
-	5.02	5	22	6.5

Operating Conditions:

Feed Temperature = 22°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 22.6 L

Distillate Feed Conditions:

Phenol Concentration = 7.1%

pH = 3.0

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0	-	-	0
-	3	-	-	5

Operating Conditions:

Feed Temperature =

Feed Flowrate = 0.75 Lpm

Bed Volume = 22.6 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Column #4 of 4 columns in series.
- A composite sample of the effluent was taken over the 3 hour regeneration step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Ma. 9, 1990

Task 2.6 - Run #: 1

Cycle #: 1/1

Operator: Dan Voth

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.5	4	21	
-	1	4	21	
-	1.5	4	21	
-	2	3	21	

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.79 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 7.1%

pH = 3.0

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.3	-	20	8
-	0.6	-	20	8
-	0.9	-	22	8
-	1.2	-	22	8
-	1.5	-	25	8
-	1.8	-	25	20
-	2.1	-	26	10
-	2.4	-	27	8

Operating Conditions:

Feed Temperature = 48°C

Feed Flowrate = 0.26 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 50% by volume.
- A composite sample of the effluent was taken over the 2 hour loading step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- After an elapsed regeneration time of 1.8 hours, the effluent sample had a distinct phenol layer.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Ma. 8, 1990

Task 2.6 - Run #: 2

Operator: Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.5	4	21	
-	1	4	21	
-	1.5	4	21	
-	2	3	21	

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.2	-	20	8
-	0.4	-	20	8
-	0.6	-	22	8
-	0.8	-	25	8
-	1	-	25	25
-	1.2	-	26	8
-	1.4	-	26	5
-	1.6	-	29	1

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.79 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 7.1%

pH = 3.0

Operating Conditions:

Feed Temperature = 48°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 50% by volume.
- A composite sample of the effluent was taken over the 2 hour loading step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- After an elapsed regeneration time of 1.0 hours, the effluent sample had a distinct phenol layer.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: Ma. 7, 1990 **Task 2.6 - Run #:** 3 **Operator:** Dan Voth

Cycle #: 1/1

Loading Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.5	4	21	
-	1	4	21	
-	1.5	4	21	
-	2	3	21	

Regeneration Step

Time	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
-	0.15	5	20	8
-	0.3	4	20	8
-	0.45	4	20	29
-	0.6	4	23	8
-	0.75	4	28	5
-	0.9	4	29	3
-	1.05	3	31	1
-	1.2	3	31	1

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.79 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 7.1%

pH = 3.0

Operating Conditions:

Feed Temperature = 51°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 50% by volume.
- A composite sample of the effluent was taken over the 2 hour loading step.
- 100 mL samples were taken during loading and regeneration steps after discharging first 100 mL.
- After an elapsed regeneration time of 0.45 hours, the effluent sample had a distinct phenol layer.

* *These phenol concentrations were neglected due to possible sample contamination.*

LOG SHEET

Date: May - June, 1990 **Task 3.0 - Run #:** 1 **Operator:** Dan Voth

Cycle #: 1 - 11

Loading Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
1	2.5			1.74
2	2.5			1.62
3	2.5			1.7
4	2.5			1.98
5	2.5			0.82
6	2.5			-
7	2.5			-
8	2.5			-
9	2.5			4.9 *
10	2.33			3.1 *
11	2.17			-

Regeneration Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
1	1			8
2	1			6
3	1			6
4	1			8
5	1			8
6	1			-
7	1			-
8	1			-
9	1			-
10	1			-
11	1			-

Operating Conditions:

Feed Temperature = 21°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 5.5%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 50% by volume.
- Except where indicated, phenol concentrations are given for composite samples.
- For cycle #11 rinsing step, residual formaldehyde concentrations were as follows:

>30% after 5 mins	7.3% after 15 mins
15.8% after 10 mins	1.2% after 20 mins

* *These phenol concentrations were for samples taken at the end of the operating step.*

LOG SHEET

Date: June, 1990

Task 3.0 - Run #: 1

Operator: Dan Voth

Cycle #: 12 - 26

Loading Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
12	2.17			2.5 *
13	2			-
14	2			-
15	2			-
16	2			-
17	2			-
18	2			0.95 *
19	2			-
20	2			-
21	2			-
22	2			-
23	2			-
24	2			-
25	2			0.8
26	2			0.8

Regeneration Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
12	1			4 *
13	1			-
14	1.17			-
15	1.17			-
16	1.17			-
17	1.17			-
18	1.17			1 *
19	1.17			-
20	1.17			-
21	1.17			-
22	1.17			-
23	1.17			-
24	1.17			-
25	1.17			1 *
26	1.17			1 *

Operating Conditions:

Feed Temperature = 22°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 5.0%

pH = 2.8

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 50% by volume.
- Except where indicated, phenol concentrations are given for composite samples.
- Rinsing step extended to 20 minutes.

* *These phenol concentrations were for samples taken at the end of the operating step.*

LOG SHEET

Date: Jul 4 - 17, 1990

Task 3.0 - Run #: 2

Operator: Dan Voth

Cycle #: 27 - 43

Loading Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
27	2			0.73
28	2			-
29	2			0.86
30	2			-
31	2			0.82
32	2			-
33	2			0.73
34	2			-
35	2			0.73
36	2			-
37				0.78
38	2			-
39	2			1.4
40	1.92			0.25 *
41	1.92			0.6
42	1.92			-
43	1.92			0.6

Regeneration Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
27	1.17			4
28	1.17			-
29	1.17			4
30	1.17			-
31	1.17			4
32	1.17			-
33	1.17			5
34	1.17			-
35	1.17			4
36	1.17			-
37	1.17			4
38	1.17			-
39	1.17			4
40	1.17			-
41	1.17			4
42	1.17			-
43	1.17			4

Operating Conditions:

Feed Temperature = 23°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 3.5%

pH = 2.9

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 50% by volume.
- Except where indicated, phenol concentrations are given for composite samples.
- Rinsing step extended to 20 minutes.

* *These phenol concentrations were for samples taken at the end of the operating step.*

LOG SHEET

Date: Jul 18 - 20, 1990

Task 3.0 - Run #: 3

Operator: Dan Voth

Cycle #: 44 - 50

Loading Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
44	1.92			4
45	1.83			1.2
46	1.75			0.72 *
47	1.75			0.84
48	1.75			-
49	1.75			0.98
50	1.75			0.98

Regeneration Step

Cycle #	Elapsed Time (hrs)	Feed Pressure (psig)	Effluent Temp. (°C)	Effluent Phenol Conc. (%)
44	1.17			8
45	1.33			11
46	1.33			10
47	1.33			11
48	1.33			-
49	1.33			11
50	1.33			11

Operating Conditions:

Feed Temperature = 23°C

Feed Flowrate = 0.39 Lpm

Bed Volume = 9.4 L

Distillate Feed Conditions:

Phenol Concentration = 8.3%

pH = 5.8

Operating Conditions:

Feed Temperature = 50°C

Feed Flowrate = 0.52 Lpm

Bed Volume = 9.4 L

Formaldehyde Feed Conditions:

Formaldehyde Concentration = 40%

pH = 2.8 - 4

Comments: *(Please indicate average sample volume for Loading and Regeneration steps; observations on formaldehyde cloudiness and other liquid or bed discolorations; extent of bed expansion during upflow; swelling or contraction of bed in different feeds)*

- Two columns in series, each packed with resin to 50% by volume.
- Except where indicated, phenol concentrations are given for composite samples.
- Rinsing step extended to 20 minutes.

* *These phenol concentrations were for samples taken at the end of the operating step.*

APPENDIX C

Pilot Plant Drawings

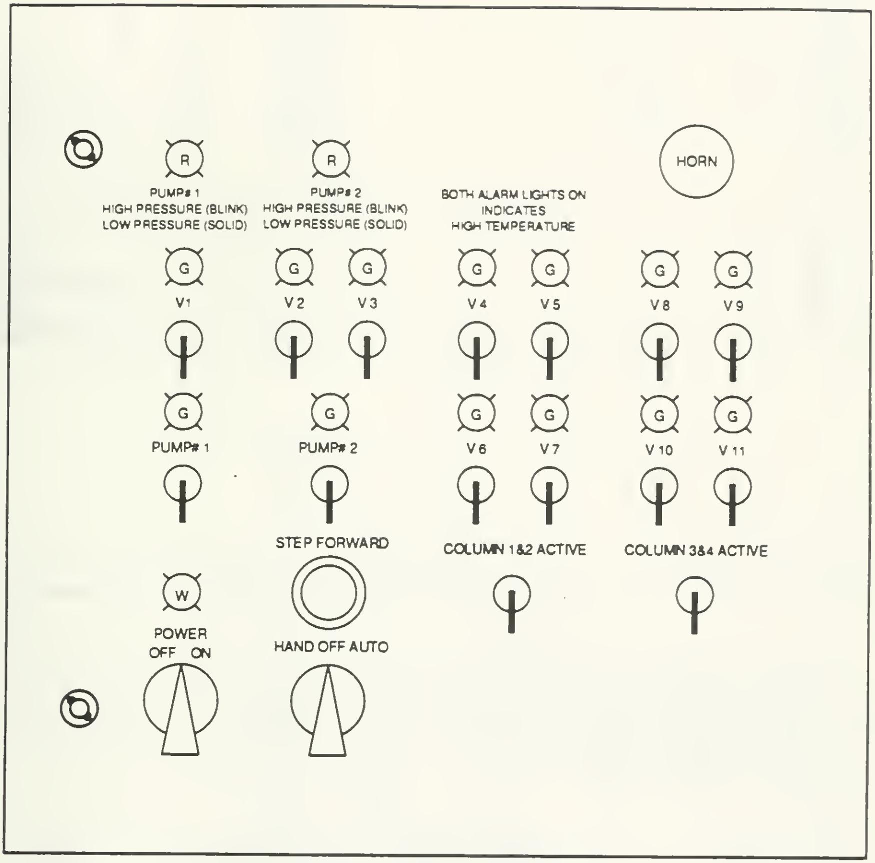
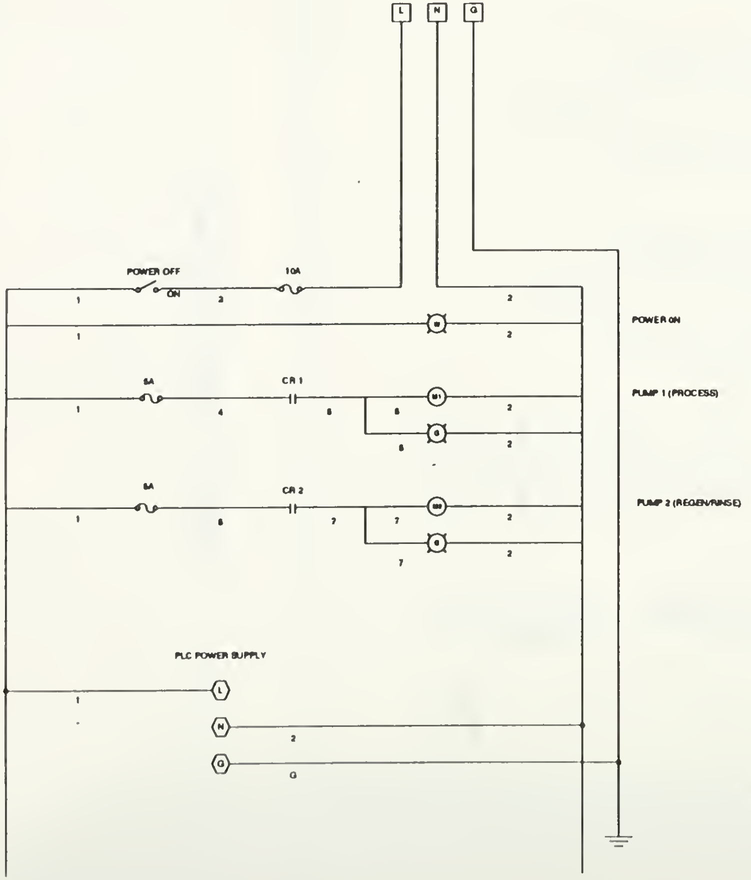
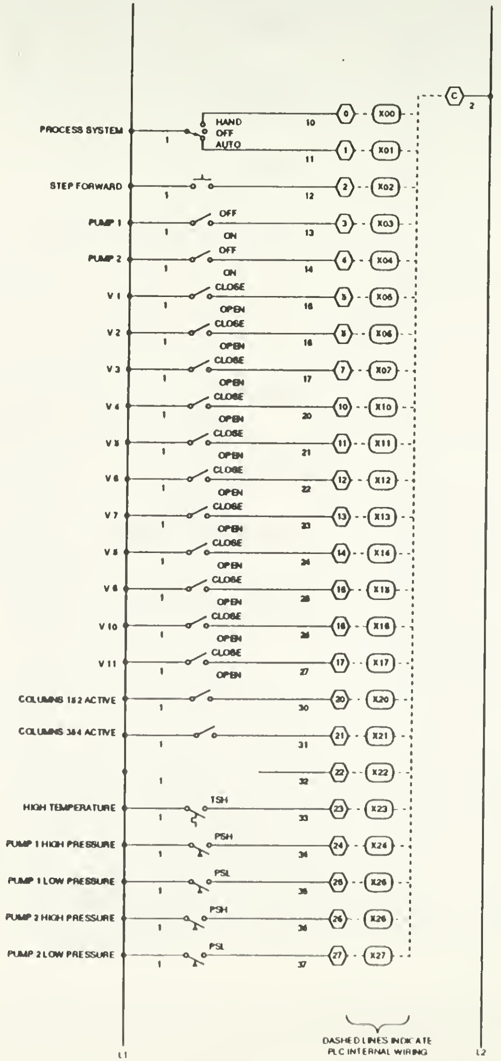


Figure C.1 - Control Panel Layout



REVISION#	DATE	DESCRIPTION	BY	APPROVED

SCALE NONE	DRAWN A Chong DATE 1989 July 14	CHK'D DATE	APP'D DATE
CUSTOMER			CONTRACT# CO5944
TITLE Wiring Diagram Electrical			DRAWING# Figure C



nn INDICATES TERMINAL BLOCKS IN PLC

X nn INDICATES PLC INPUT# FOR PROGRAMMING PURPOSES

Y nn INDICATES PLC OUTPUT# FOR PROGRAMMING PURPOSES

XX INDICATES TERMINAL BLOCKS FOR CONNECTING TO CUSTOMER'S EQUIPMENT

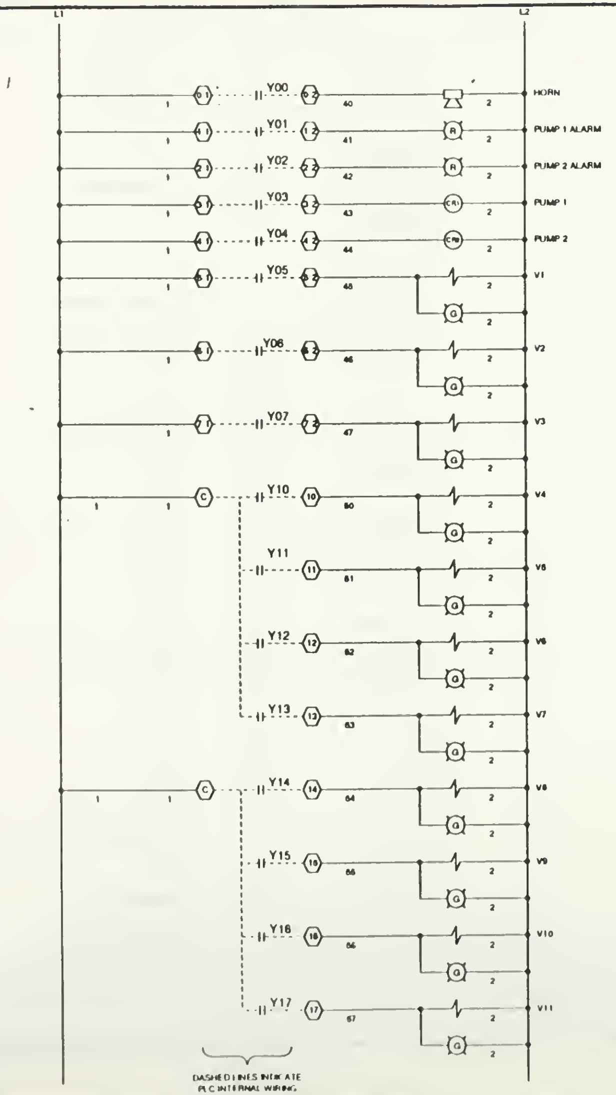
ALL PRESSURE SWITCHES N O CONTACTS CLOSE WHEN PRESSURE ABOVE SETPOINT

REFERENCE DRAWINGS

PROCESS AND INSTRUMENTATION DIAGRAM
PANEL LAYOUT
WIRING DIAGRAM PAGE 1 (ELECTRICAL)
WIRING DIAGRAM PAGE 2 (INPUTS PLC)
WIRING DIAGRAM PAGE 3 (OUTPUTS PLC)

REVISION#	DATE	DESCRIPTION	BY	APPROVED

SCALE NONE	DRAWN A Chong DATE 1989 July 22	CHK'D DATE	APP'D DATE
CUSTOMER			CONTRACT # [illegible]
TITLE Wiring Diagram PLC Inputs			DRAWING # [illegible]



nn INDICATES TERMINAL BLOCKS IN PLC

X nn INDICATES PLC INPUT# FOR PROGRAMMING PURPOSES

|| Ym INDICATES PLC OUTPUT# FOR PROGRAMMING PURPOSES

XX INDICATES TERMINAL BLOCKS FOR CONNECTING TO CUSTOMER'S EQUIPMENT

REFERENCE DRAWINGS

PROCESS AND INSTRUMENTATION DIAGRAM
PANEL LAYOUT
WIRING DIAGRAM PAGE 1 (ELECTRICAL)
WIRING DIAGRAM PAGE 2 (INPUTS PLC)
WIRING DIAGRAM PAGE 3 (OUTPUTS PLC)

REVISION#	DATE	DESCRIPTION	BY	APPROVED

SCALE NONE	DRAWN A Chong DATE 1989 July 14	CHK'D DATE	APP'D DATE
CUSTOMER			CONTRACT # [illegible]
TITLE Wiring Diagram PLC Outputs			DRAWING # Figure 4

APPENDIX D

Manufacturer's Literature on Amberlite XAD7

table I—typical properties of Amberlite XAD-7

Appearance	Hard, hydrated opaque beads
Average particle diameter	0.30 to 0.45 mm
Harmonic mean particle size	0.45 to 0.60 mm
Solids	28% to 33%
True wet density, in distilled water°	1.05 g/ml
Skeletal density	1.24 g/ml
Bulk density	41 lbs/ft³ (656 g/l)
Pressure drop, in water	See Figure 1
Hydraulic expansion during backwash	See Figure 2

The following properties were determined for dried Amberlite XAD-7. The values for solvated beads will be different owing to the swelling of the gel phase upon solvation. The resultant pore enlargement, particularly in aqueous systems, allows for adsorption of very large molecules.

Porosity of beads	0.50 to 0.55 milliliter of pore/ milliliter of bead.
Surface area	450 m²/g
Average pore diameter	80 Ångstrom units

°The density of Amberlite XAD-7 in any system is directly proportional to the density of the solvent being used.

process of adsorption

To better understand the phenomena through which Amberlite XAD-7 functions, it is helpful to consider the individual beads each to be an agglomerate of a large number of very small microspheres as shown on the following sketch.

The macrorecticular structure results in a continuous solid phase as well as a continuous pore phase. The pores fill with solvent during use, exposing a large surface area of adsorbent to the solvent and to the solute molecules. This results in rapid diffusion of solvent and solute through the adsorbent and, hence, excellent adsorptive rate properties.

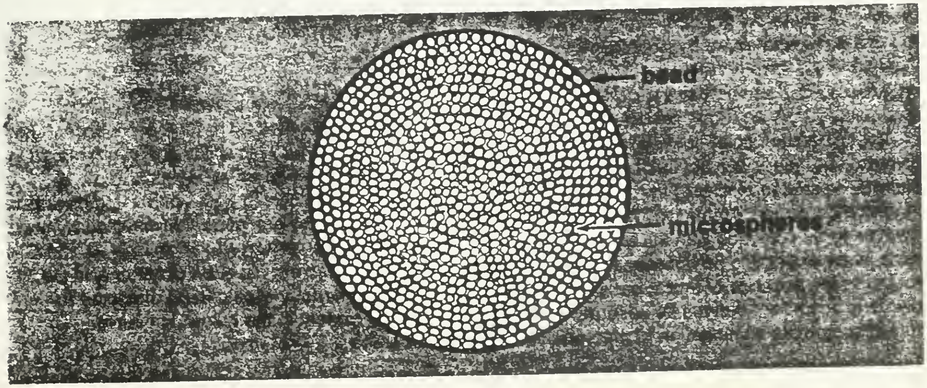
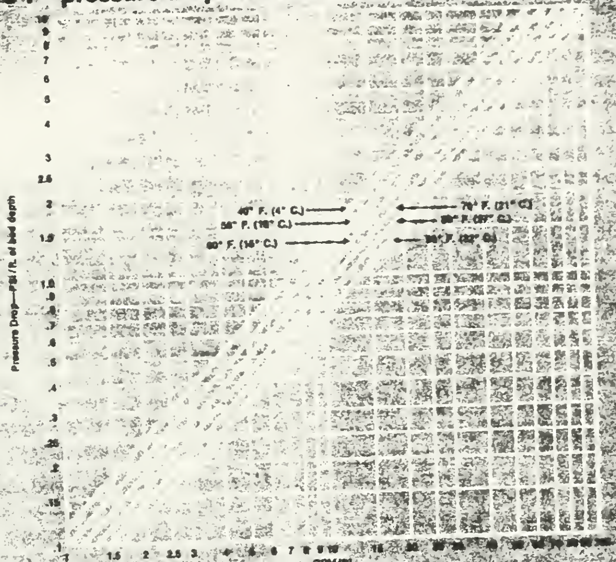
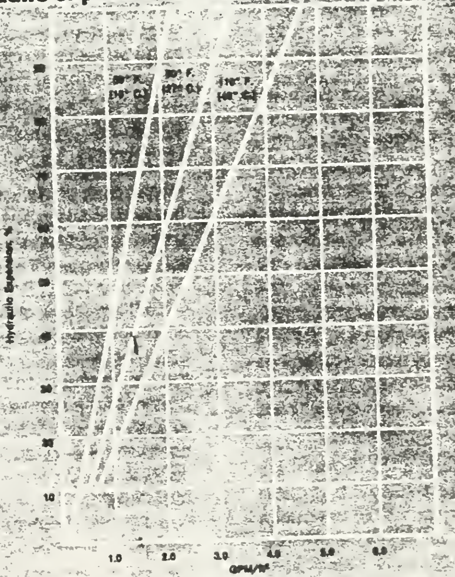


figure 1—pressure drop vs water flow rate—Amberlite XAD-7



Metric Conversion
GPM/ft² to M/hr = GPM/ft² × 2.45
PSI/ft to MH₂O/M resin = PSI/ft × 2.30

figure 2—hydraulic expansion vs water flow rate—Amberlite XAD-7



Metric Conversion
GPM/ft² to M/hr = GPM/ft² × 2.45